JULY 1951

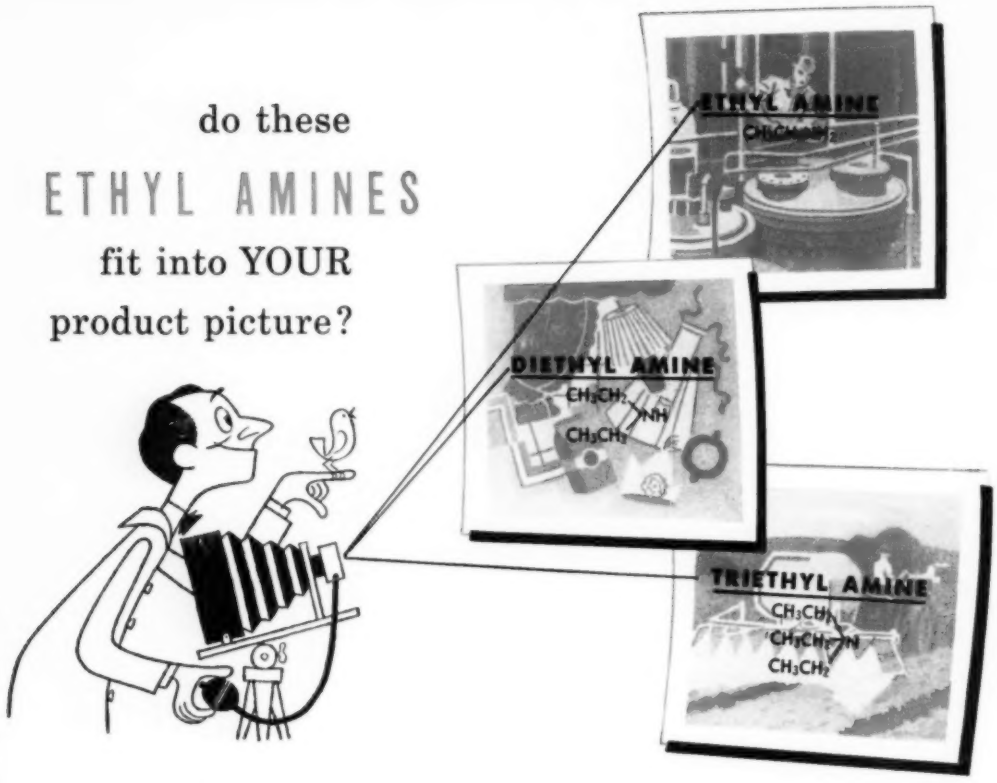
Chemical Engineering Progress

PUBLISHED MONTHLY BY THE AMERICAN INSTITUTE OF CHEMICAL ENGINEERS

do these

ETHYL AMINES

fit into YOUR product picture?



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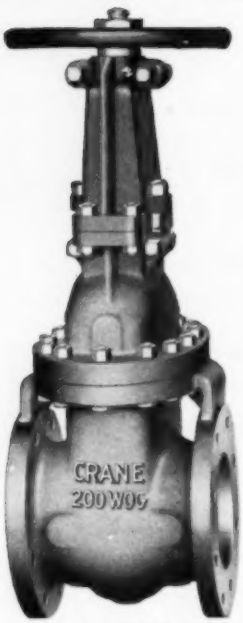

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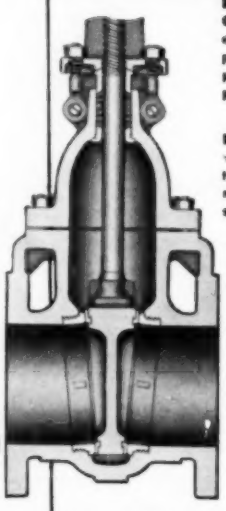

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Technical Director

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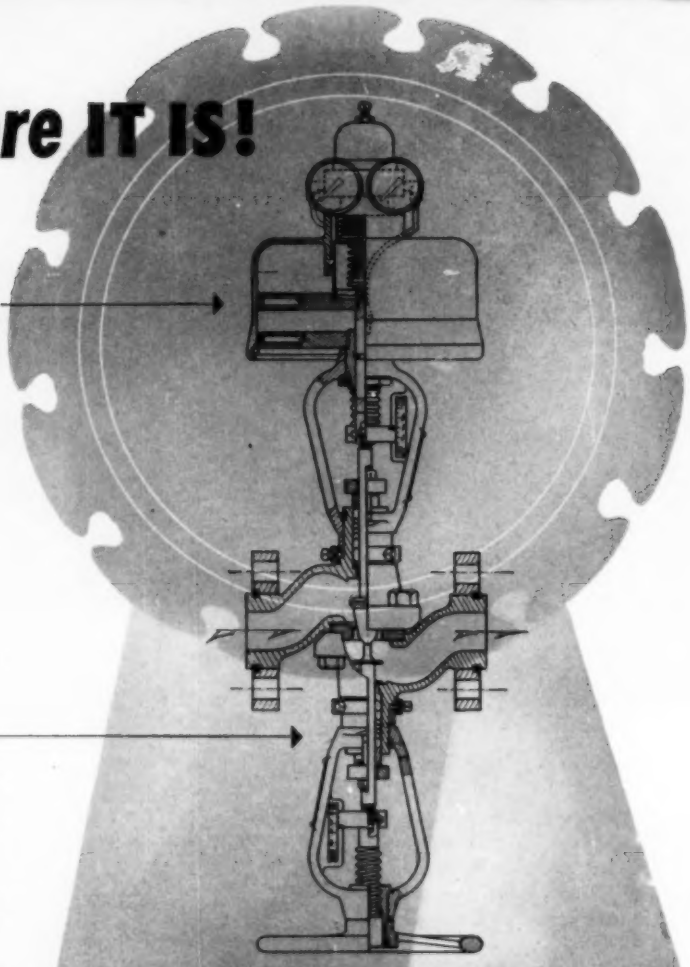

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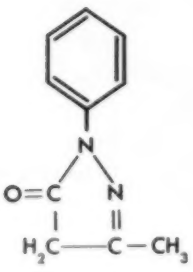

PROPERTIES	
White to slightly yellow powder	
Boiling point at 17 mm. Hg	191°C.
Melting point	128.9°C.
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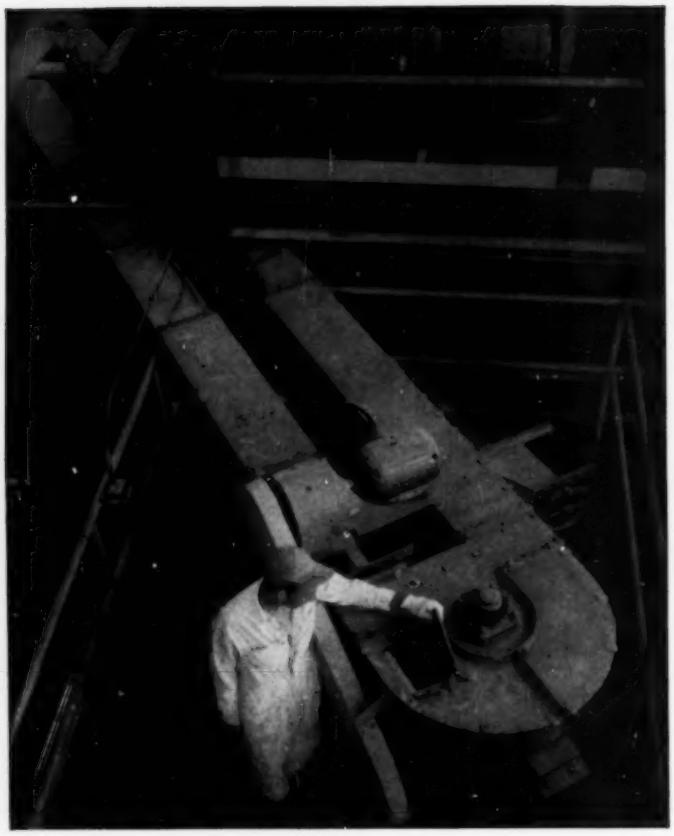

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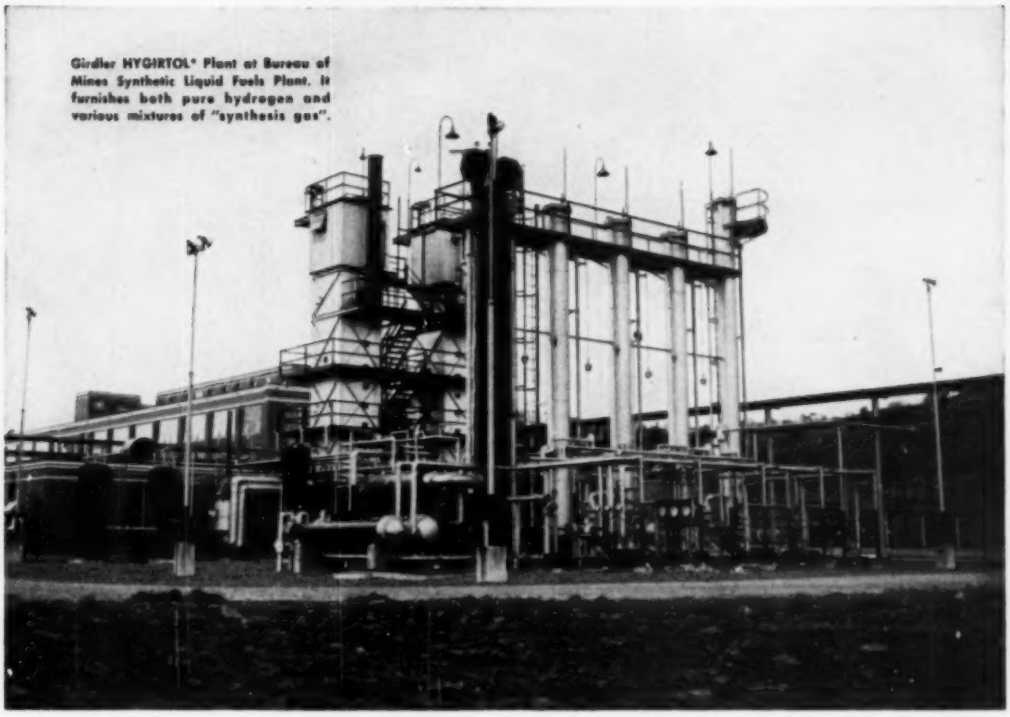

Girdler HYGIRTOL* Plant at Bureau of Mines Synthetic Liquid Fuels Plant. It furnishes both pure hydrogen and various mixtures of "synthesis gas".

Girdler gas plant speeds synthetic fuels process development

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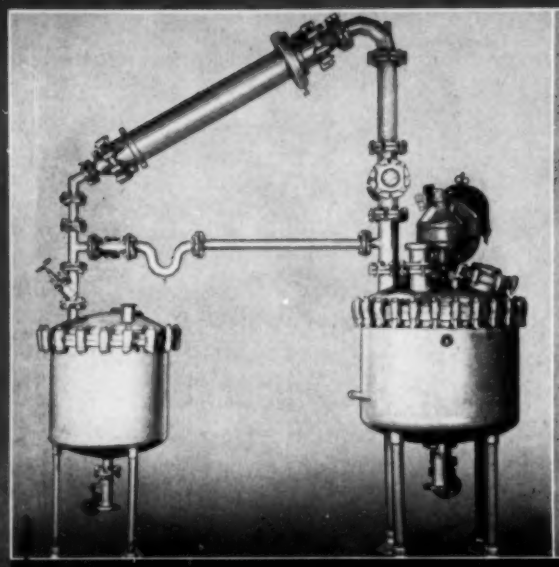
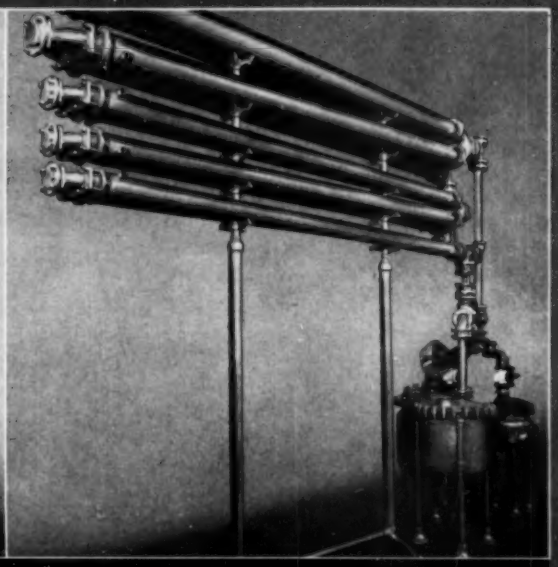
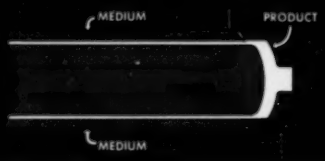
Gas Processes Division





Double Jacketed Glass-Lined Condensers employ internal and external jackets for maximum cooling capacity per square foot of surface.



Jacketed Glass-Lined Pipe with glass-lined return bends are effective for cooling or heating, especially under pressure.

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OPINION AND COMMENT

THE CASE FOR GOVERNMENT RESEARCH CONTRACTS

MANY companies now face a troublesome question: to take or not to take government research contracts.

Such a contract has certain obvious disadvantages. It means entanglement with the red tape of the government bureaus and the armed forces; it often calls for dealing with people who have little understanding of research; it exposes the company, after the emergency is over, to the unjustified charge by demagogues that industry has used public funds to acquire patents and other advantages; because of the military security involved it complicates research administration and hampers the exchange of technical ideas within the research department; and, most important, it diverts precious manpower and facilitates away from the primary objectives of the organization.

For the technical employee, a research contract with the government has some of the same disadvantages it has for the company. Security regulations, for example, are a personal annoyance; they are also undesirable because they necessarily reverse the industry trend toward freer publication of research results.

These disadvantages to the company and to the individual research man cannot be denied. However, the benefits to the nation arising from government research contracts with industry are substantial. The basic point, in the author's opinion, is that industry can do virtually any research job better, quicker, and cheaper than government can. There are a number of reasons. Industry research is more efficient than government research. Competition is as severe in research as in other segments of the industrial system, and it weeds out the less effective of the industrial organizations. This beneficial effect of competition is not available to government research.

In striving for a competitive position, industrial research has sought out and secured highly competent research administrators. The government is handicapped by shortsighted policies on salaries to be paid professional men, and attempts to operate with less talented personnel. When the Office of Naval Research sent a questionnaire to scientists leaving government positions, one of the principal areas of dissatisfaction was found to be the poor quality of the technical administration, particularly at the higher levels.

The demands of the defense program require a great increase in military research and development effort. If industry is not prepared to accept its share of this load, the alternative may be the creation of large new, hastily organized government laboratories. This step would inevitably drain men from industry, where they have been functioning effectively, and so would be wasteful of scientific manpower. Industry has at its disposal competent research teams, which might be directed to the solution of the more important defense research projects. These teams should be maintained intact, so that they may work with maximum effectiveness.

While the national interest represents the chief incentive to industry to take government research contracts, there are incidental benefits. A number of research men in industry are subject to reserve call or to the draft, and these men are important in a time of shortage. Government research contracts may help to hold these key men in industry. War in Europe might create hysteria to draft technical men, but there seems a good chance that better sense would prevail. At a time when we may have to fight a highly technical war, the draft obviously ought to leave intact research teams working on chemicals, petroleum products, electrical equipment, and many other items. These are areas in which the country cannot afford to be wrong. In the last war, we were wrong too often. As just one example, poor design of American torpedoes caused the failure of many of them to explode when they struck Japanese ships. There is no way to determine accurately the harm that comes from poor-quality weapons and matériel. On the average, however, when an agency puts a scientist or engineer into a position not requiring his full professional abilities, it may be signing the death warrant of a number of American soldiers and sailors, who are thereby forced to use less effective weapons or equipment. The engineer and the research scientist who wish to make their maximum contribution to the defense effort will stay in industry, however much they would like to get into the actual fighting.

When all these factors are considered, it appears that industry is well advised to accept a reasonable amount of government work. This is true even if departments must be expanded to handle government contracts. Companies should look ahead five or ten years, and try to decide what their future requirements will be. Prompt expansion up to those limits (to the extent that men are available) represents a most efficient method of aiding the national defense, and one that will place industry in a strong position to serve the economy after the emergency is over.

Government contracts may involve headaches for research administrators now, and undeserved brickbats for the company later. There will be more than compensating desirable results, however, as regards technical personnel, the immediate national safety, and the long-range public welfare.

R. C. Gunness

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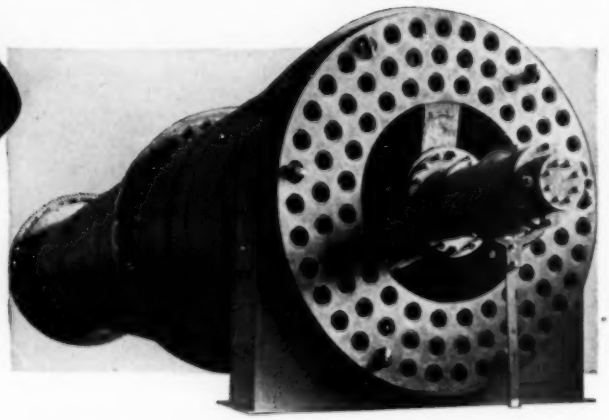

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PRINCIPLES OF REACTOR DESIGN

HOMOGENEOUS REACTIONS

J. R. CADDELL and D. M. HURT

Du Pont Company, Wilmington, Delaware

For a generally useful development of reactor design principles, classification of reactors on the basis of the number and type of reacting phases involved is shown to be more satisfactory than the earlier unit process concept.

A simplified method is presented for (1) correlating kinetic data of homogeneous reactions in order to calculate optimum reaction conditions from a minimum amount of experimental data, and (2) applying such rate data in the design of homogeneous reaction equipment. This procedure intentionally omits mathematical exactitude with respect to minor variables to the extent consistent with the accuracy required for engineering design. Simplification is obtained by: (a) use of the number-of-reaction-units concept (a dimensionless number including reaction rate constant) as a measure of reactor performance. This concept, previously employed only with first-order reactions, has been extended to cover other cases encountered in industry, (b) use of identical equations for both constant-pressure and constant-volume reactions, and (c) use of graphical solutions of kinetic rate equations to avoid repetitive calculations.

THE rational design of chemical reactors presents one of the most challenging problems in the field of chemical engineering. Today's highly competitive economy demands much more than a mere multiplication of laboratory units when expanding to plant capacity. Reactor design, in its broadest sense, thus must include the determination, by comparative evaluations, of the economic optimum type, size, and shape of equipment for any specified capacity and performance.

In any general study of reactor design principles, it is desirable to exploit broad principles of similarity. It has been our experience that classification of reactors according to the apparent order of reaction and according to the number of reacting phases provides greater similarity than the earlier unit-process concept. The phase classifications for all reactions and reaction equipment are given in Table 1. Classification of reactions is based on reactants only. For purposes of definition, all catalysts are considered as reactants; thus, when all reactants (including catalysts) are in a single phase the reaction is homogeneous.

In industrial work, applied reaction kinetics must be considered solely from the standpoint of its value as a labor-saving device. In other words, the broad principles of classical kinetics are utilized primarily to minimize the time required to develop complete rate data and design information for certain specific reactions. In line with this viewpoint, simplified procedures also have been developed for analyzing reaction data and for sizing plant equipment. Mathematical exactitude with respect to minor variables is omitted to the extent consistent with the accuracy required for design.

It has been found convenient to employ the number-of-reaction-units concept as a measure of reactor performance. This term is a dimensionless number which includes the reaction rate constant of conventional rate equations. Heretofore the number-of-reaction-units concept has been applied only to first-order reactions. In the current work this concept is extended to cover other cases met in industrial practice. The number-of-reaction-units concept is broadly analogous to the transfer-unit concept used in diffusional rate processes. The number of reaction units is numerically proportional to the size of equipment required.

Application of reaction kinetics principles has been simplified by using identical equations for both constant-volume and constant-pressure reactions. Complex equations are avoided by treating

TABLE 1.—CLASSIFICATION OF REACTIONS

Based on Reactants Plus Catalysts But Not Products

Homogeneous	Heterogeneous	
1. Gas phase	1. Gas-solid phases	4. Liquid-liquid phases
2. Liquid phase	2. Gas-liquid phases	5. Solid-solid phases
3. Solid phase	3. Liquid-solid phases	6. Gas-liquid-solid phases

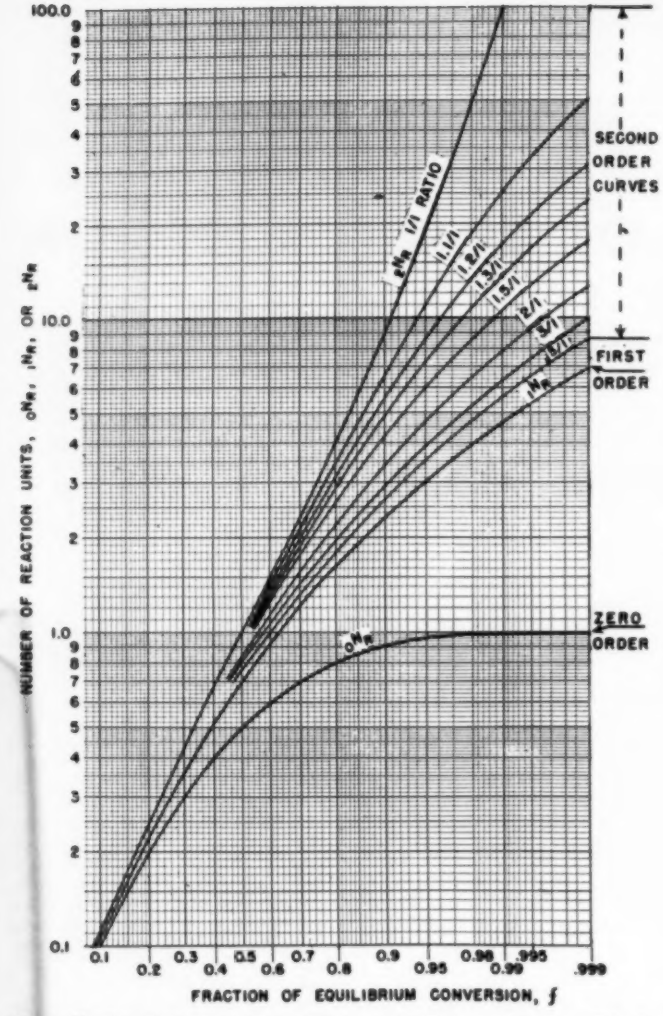


Fig. 1. Number of reaction units vs. fraction of equilibrium conversion. This chart is a graphical solution of the rate equations given in Table 2. A detailed procedure for using these curves is given in Table 3. Where two reactants are present, f refers to the one present in lower concentration. For second-order equations, a family of curves is given for various possible ratios of initial reactant concentrations.

the actual conversion as the fractional approach to equilibrium conversion. Repetitive calculations are avoided by the use of graphical solutions for the kinetic rate equations.

A brief summary is presented of the methods for: (1) correlating the kinetic data of homogeneous reactions in order to calculate optimum reaction conditions from a minimum amount of experimental data, and (2) applying such rate data in the design of homogeneous reaction equipment.

Procedures demonstrated herein for homogeneous reactions may be applied also in the heterogeneous field. However, in this latter case they apply only to the chemical, or nondiffusional component of the over-all rates.

Correlation of Test Data for Homogeneous Reactions

Experimental Check for Homogeneity. Experimental data may be treated by homogeneous methods if it can be shown that conversion, for a given time of reaction, is essentially unaffected by a change in the surface-to-volume ratio of the reactor. The surface-to-volume ratio should be varied by as large a factor as practical, and in no case should the factor be less than two, as otherwise errors in measurement of other variables may render the results inconclusive.

Apparent Order of Reactions. Knowledge of the apparent order† is used to determine the influence of operating variables so that optimum conditions for carrying out a reaction can be predicted from a minimum number of experiments. The value of such information is that of extrapolation and interpolation.

The rate equations for apparent zero-order, first-order, and second-order reactions are tabulated in Table 2. These equations can be used for reactions at constant pressure or at constant volume. Extrapolation should normally be limited to doubling the maximum or halving the minimum number of reaction units as measured experimentally. For this range of extrapolation, the relative error in neglecting volume changes is a maximum of 10% of the number of reaction units when no inert diluents are present, and decreases as inerts are added. Use of the graphical solutions of the rate equations (Fig. 1) saves appreciable time in analyzing reaction data. The procedure for obtaining corresponding values of f, N_R, and θ is given in Table 3.

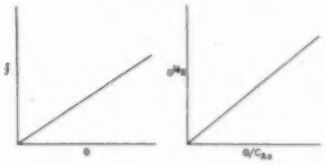
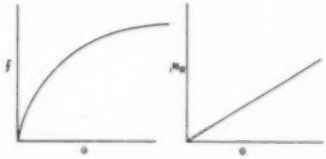
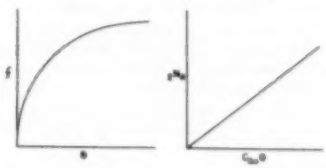
Apparent third-order and higher-order reactions are rare. While these may be treated by the same methods as lower orders, the material is considered to have insufficient general value to be included here. A commercially important reaction is the third-order reaction that occurs in the ammonia oxidation process for manufacture of nitric acid, $2NO + O_2 \rightarrow 2NO_2$. In practice this step is combined with a concurrent gas-liquid reaction and should be considered as a special case rather than a representative example.

In general, determination of reaction order provides the most information from few data, but there are three cases in which knowledge of the apparent order is not useful or essential. They are:

† The apparent order of a reaction is a classification according to the number of molecules that appear to enter into the reaction. Apparent order is determined by the relationship that exists, at a given temperature, between the rate of reaction and the concentration of reactants. The treatment of applied kinetics given in this paper is based on apparent order. This is not necessarily coincident with true order, and no knowledge of the latter is required.

TABLE 2.—KINETIC EQUATIONS FOR APPARENT ORDERS OF REACTIONS

Homogeneous Reactions

Apparent Order	Rate Equations	Characteristic Graphs	Remarks
Zero	$r = k$ $_cN_R = f$		The time required for conversion of a given fraction, f, is proportional to the initial concentration, C_{Ao}; for example, doubling the concentration doubles the time for a given f.
First	$r = kC_A$ (b) $_1N_R = \ln \frac{1}{1-f}$		The time required for conversion of a given fraction, f, is not affected by a change in the concentration.
Second Unequal concentrations $C_{Ao} < C_{Bo}$	$r = kC_AC_B$ (b) $_2N_R = \frac{C_{Bo}}{C_{Ao} - C_{Bo}} \ln \frac{1-f}{1-\frac{C_{Ao}}{C_{Bo}}f}$		a. The time required for conversion of a given fraction, f, is inversely proportional to the initial concentrations; doubling the concentrations reduces the time by one half. b. Where two reactants are present, f refers to the one present in lower concentration. The concentration of the other reactant is shown with time on the abscissa of the $_2N_R$-time plot.
Second Equal concentrations $C_{Ao} = C_{Bo}$ (a)	$r = kC_A^2$ (b) $_2N_R = \frac{f}{1-f}$		The time required for conversion of a given fraction, f, is inversely proportional to the initial concentrations; doubling the concentration reduces the time by one half.

(a) If the initial concentration, $C_{Bo} = C_{Ao}$, the reaction is kinetically similar to one in which two moles of a single reactant react to form products.

(b) Although this equation is strictly valid only for irreversible reactions, it is included in order to explain more clearly the remarks on concentration and rate of reaction. This equation is not needed in solving practical design problems by the method outlined in this paper.

TABLE 3.—PROCEDURE FOR OBTAINING CORRESPONDING VALUES OF f, N_R, AND θ

Homogeneous Reactions

This chart outlines the procedure for using the graph appearing in Figure 1 in order to obtain corresponding values of f, N_R, and θ. For purposes of example it is assumed that (1) the apparent order of reaction has been established as first order, (2) a few values of f vs. θ as needed to determine apparent order are available, and (3) it is desired to obtain complete information on corresponding values of f, N_R, and θ over the range of 0-99% conversion. In this manner both steps of going from f to N_R and N_R to f can be illustrated with one example. In a given case only part of this procedure may be pertinent depending on the data at hand.

Operation	Steps to be Taken	Remarks
1. Tabulate experimental data on conversion vs. time of reaction	See Table A below.	Experimental data are for θ and f only. For values of N_R refer to operation number 2.
2. Obtain values of N_R corresponding to the experimental values of f	Refer to Fig. 1.	For this example (apparent first order) the corresponding values are shown in Table A.
3. Plot the values of N_R vs. the appropriate function of C_{Ao} and θ. See remarks and figure below.	Refer to Table 2 for the correct function.	For this example (apparent first order) the plot is $_1N_R$ vs. θ.
4. Extrapolate the N_R curve and complete tables of f, N_R, and θ, or prepare plots of f vs. θ or N_R vs. θ.	Read values of f corresponding to given values of N_R from the chart, Fig. 1.	A sample table has been completed, Table B. See page 334 for limits of extrapolation.

TABLE A.—EXPERIMENTAL DATA

θ min.	f	$_1N_R$
0	0	0
50	.70	1.2
100	.89	2.25

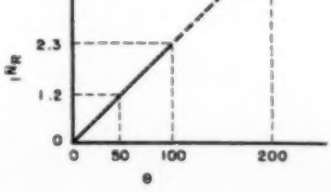


TABLE B.—CORRESPONDING VALUES OF θ, f, AND N_R

θ	$_1N_R$	f
20	0.45	.36
40	0.9	.59
60	1.35	.73
80	1.8	.83
100	2.3	.90
120	2.75	.94
140	3.2	.96
160	3.7	.975
180	4.1	.983
200	4.6	.99

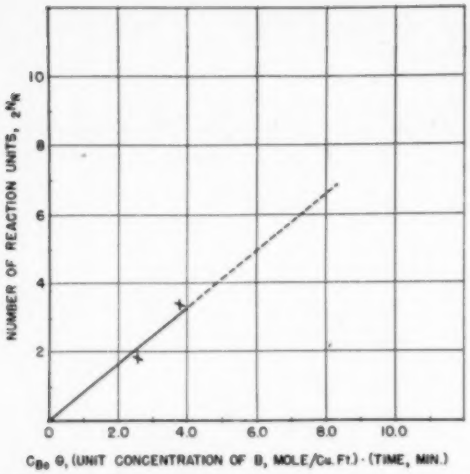


Fig. 2. Determination of apparent order for reactor design example; isothermal reaction at 50° C.

1. If the apparent order is fractional (the rate characteristics are between those for zero and first or first and second apparent orders as shown in Fig. 1), information on the apparent order is not generally useful because interpolation and extrapolation are not reliable.
2. The order of reaction need not be determined where complete rate data for all conversion levels and reactant concentrations of potential interest are already available from a large number of experiments.
3. Knowledge of the apparent order of a reaction is not essential where obvious economic considerations limit the concentration of reactants. In this case, one can obtain experimental rate data for the permissible concentration, and design directly from these.

Determination of Reaction Order. With one reactant, it is assumed that the reaction is either first or second order. Values of $_1N_B$ and $_2N_B$ corresponding to the experimental data on fractional conversion are plotted and examined for linearity. If a straight line fits the data on a plot of $_1N_B$ vs. θ, the reaction is apparent first order; if a straight line fits the data on a plot of $_2N_B$ vs. $C_{Ao}\theta$, the reaction is apparent second order. If a straight line fits the data on either plot equally well, the data are inconclusive, or the apparent order may be fractional.

With two reactants, it is assumed that the reaction is second order, and the values of $_2N_B$ are read from the appropriate curve (Fig. 1) corresponding to the ratio of initial concentrations. If a straight line fits the data on a plot of $_2N_B$ vs. $C_{Bo}\theta$ (where the concentration of reactant B is greater than A, *see* Table 2), the reaction is second order with each component having a first-order effect. If a straight line does not fit, closer examination is required. It is then necessary to have data on the conversion of each reactant while the other is in large excess. These results on conversion are then examined as for a single reactant. The reaction may be first or second order with respect to either reactant.

Multiple Reactions. There are two types of multiple reactions, *consecutive* and *simultaneous*, and it is important to distinguish between these in order to obtain optimum ratios of products through proper variation of reaction conditions. With multiple reactions, the reaction time (equipment hold-time) depends on the events that result from the several reactions. Complete treatment is not given in this paper, but major considerations are discussed qualitatively.

In consecutive reactions the original reaction is followed by one or more subsequent reactions in which the products first formed decompose or react with each other or react with the feed to form a new set of final products. The chlorination of methane is an example.

$$CH_4 + Cl_2 = CH_3Cl + HCl \quad (1)$$

$$CH_3Cl + Cl_2 = CH_2Cl_2 + HCl \quad (2)$$

$$CH_2Cl_2 + Cl_2 = CHCl_3 + HCl \quad (3)$$

$$CHCl_3 + Cl_2 = CCl_4 + HCl \quad (4)$$

The ratio of products is a function of (1) the feed gas composition, (2) time, and (3) the rates of the individual reactions. Individual reaction rates are obtained from a series of separate tests with each intermediate. For example, in the chlorination of methane, the rate of reaction 2 is found by reacting a large excess of CH_3Cl with little chlorine. Under these conditions, the chlorine is used almost entirely to form CH_2Cl_2; the reaction is apparent first order (with respect to chlorine), and the rate can be determined. Having obtained rate data for each individual reaction a general solution may be made (involving somewhat complex mathematics) or specific solutions of interest may be evaluated through relatively simple (but somewhat lengthy) stepwise calculation.

In simultaneous reactions the reactants are capable of reacting in more than one way to form different products, and several reactions proceed simultaneously. The esterification of phthalic anhydride and butyl alcohol with acid catalysts is an example.

$$C_6H_4(CO)_2O + 2C_4H_9OH \rightarrow C_6H_4(COOC_4H_9)_2 + H_2O$$

$$xC_6H_4(CO)_2O + yC_4H_9OH \rightarrow \text{Tars (color-formers)} \quad (5)$$

Each of the several possible reactions has its own characteristic rate, one usually being much faster than the others. The total rate of disappearance of the reactants is the sum of the rates of the individual reactions. The ratio of products is a function of (1) time and (2) the individual reaction rates. If the separate reactions are of different order, the ratio of products will also be a function of the concentration (partial pressure) of the reactants. It should be noted that, in general, the ratio of products from simultaneous reactions is not dependent on the feed composition. Observation of the effect of feed gas composition may be used in most cases to determine whether the reactions are simultaneous or consecutive.

The desired ratio of products may be obtained by varying either (1) time of reaction, (2) concentration of reactants, (3) temperature of reaction, or (4) by

TABLE 4.

Concentration of Reactants mole/cu.ft. of solution		Reaction Time, θ, min.	Fraction of Equilibrium Conversion, f, with Respect to A
C_{Ao}	C_{Bo}		
0.05	0.15	0	0 *
0.05	0.15	17	0.79
0.05	0.075	52	0.86

* These values are given to show that the origin is a "free" point in plots of N_B vs. θ.

TABLE 5.

Ratio of Reactant Concentrations C_{Bo}/C_{Ao}	Value of f, the Fraction of Equilibrium Conversion with Respect to Reactant A	Value of $_2N_R$ Corresponding to Values of f
3/1	0.79	1.9
1.5/1	0.86	3.4

using homogeneous or heterogeneous catalysts. The time and temperature should be checked first. It may be that the individual reactions have widely different temperature coefficients. If the desired ratio of products can not be obtained easily by varying time, temperature, or concentration, the use of catalysts should be explored.

Effect of Temperature on Reaction Rate. Rates of chemical reactions increase or decrease with corresponding changes in the temperature at which the reactions are carried out. In general, homogeneous reactions are run at the highest temperature consistent with the dictates of corrosion, equilibrium, side reactions, and the cost of heat. Operation at any lower temperature results in a slower reaction and reduced productivity. Thus, as temperature changes, plots of f, the fraction of equilibrium conversion vs. time will also change. Note that, if the equilibrium should decrease materially, the fraction of equilibrium conversion corresponding to a given number of reaction units might actually represent the production of a smaller amount of material.

Data on the rate of reaction at different temperatures are used (1) as the basis for an economic balance between the size of the reactor and the cost of reaching and maintaining the temperature of the reaction; (2) to determine the optimum balance between size of reactor and conversion per pass; (3) in the case of reversible reactions to determine, in addition to the foregoing, the economic optimum operating temperature. The temperature of reaction affects both the rate of reaction and the equilibrium.

The rate of reaction is related to temperature by the Arrhenius equation,

$$k = Ae^{-E/RT} \qquad (a)$$

In this paper the following form involving the number of reaction units, N_R, is used to express the effect of temperature on the rate of reaction:

for apparent first-order reactions,

$$\frac{{}_1N_R}{\theta} = Ae^{-E/RT} \qquad (b)$$

for apparent second-order reactions,

$$\frac{{}_2N_R}{C_{Bo}\theta} = Ae^{-E/RT} \qquad (c)$$

Application to Plant Design

Application of kinetic information is illustrated with the design of homogeneous reaction equipment. The accompanying example shows the use of reaction kinetics in sizing equipment; no economic calculations are detailed but the results of such evaluations are stated where necessary to this example.

Statement of Problem

A plant process is discharging 1000 lb./hr. of an aqueous solution containing 0.05 mole/cu.ft. of material A. It is desired to convert this waste into a useful product, C, by reacting A with material B, to form the product, C. Apparent economic considerations indicate that A should be reacted at the available concentration. The addition of B has negligible effect on the volume. The specific volume of the solution is 0.12 gal./lb. Reaction temperature is limited to 50° C. because material A is temperature-sensitive. As the first step in an evaluation of alternative designs, it is desired to calculate the size of (1) a batch kettle, (2) a pipe-line reactor, (3) a single continuous kettle, and (4) each kettle in a series of three equal-volume cascade kettles. A conversion of 98% is to be obtained, and the reaction is essentially irreversible.

Experimental Data. Some data from batch operations † as shown in Table 4, are available for the reaction of A and B at 50° C.

† A similar treatment would be used if these data were from a pipe-line reactor.

TABLE 6.

Reaction Time, θ, Min.		Number of Reaction Units with Respect to Reactant A	Fraction of Equilibrium Conversion, f (from curve, Fig. 3, for 2/1 ratio)
C_{Bo} 1.0 mole/cu.ft.	C_{Bo} 0.1 mole/cu.ft.		
0.5	5	0.4	0.30
1.0	10	0.83	0.50
1.5	15	1.25	0.63
2.0	20	1.65	0.72
3.0	30	2.5	0.835
4.0	40	3.3	0.895
5.0	50	4.1	0.933
6.0	60	4.95	0.958
7.0	70	5.8	0.972
8.0	80	6.6	0.984
9.0	90	7.4	0.988

Development of Kinetic Data.

1. Assume that the reaction is second order and prepare a plot as shown in Figure 2. The fractional conversion and values of $_2N_R$ are always referred to the component of lowest concentration (A in this case); concentration of the other reactant (B) is then plotted with θ on the abscissa. A straight line fits these data thus confirming the assumption that the reaction is apparent second order. The values of $_2N_R$ corresponding to the experimental data are shown in Table 5.

2. Determine the concentration of reactant B that is to be used. In this example, an economic evaluation of the costs of separating the product C from unreacted B indicates that the optimum corresponds to feeding B to yield an initial concentration of 0.10 mole/cu.ft. of solution.

3. Use Figure 2 for interpolation and extrapolation to obtain several values of $_2N_R$ vs. θ for the economic ratio of reactants (2/1 in this case). Corresponding values of f are obtained from Figure 1. These values

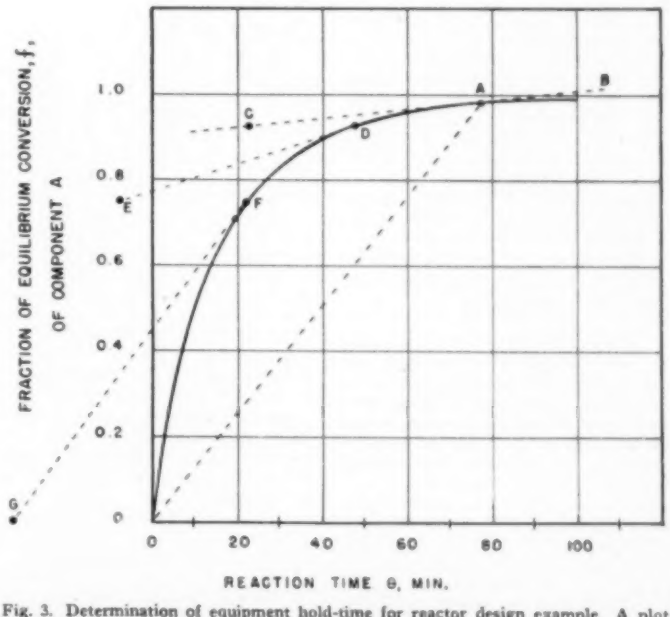


Fig. 3. Determination of equipment hold-time for reactor design example. A plot of conversion vs. reaction time in batch equipment at 50° C. and with C_{Ao} = 0.05 mole/cu.ft. and with C_{Bo} = 0.10 mole/cu.ft.

are given in Table 6 and are plotted in Figure 3; this curve is then valid for the specified temperature and concentrations.

Sizing a Batch Kettle. The curve, Figure 3, is an expansion (and smoothing) of the original data (batch). It may be used within the normal limits of extrapolation to determine, directly, the time for reaction when essentially isothermal conditions prevail.‡ The rate of reaction is a maximum at the start of the reaction when the concentration of the reactants is greatest. The rate falls off throughout the course of the reaction and is a minimum at the end of the cycle. As may be seen by inspection of Figure 3, at any one instant the rate of reaction is the slope of the tangent to the curve at the point corresponding to the instantaneous conversion level. The average rate throughout the course of the reaction is thus the slope of the chord connecting the origin and discharge point on the curve (Fig. 3).

The reaction time is read directly from the curve (Fig. 3) as 77.5 min. for 98% conversion ($f = 0.98$).

Volume of kettle or pipe-line

$$= \frac{77.5}{60}(1000)(0.12) = 155 \text{ gal.}$$

This is the volume required for reaction only; plant units must be sufficiently larger to allow for such items as freeboard, heat transfer, and time for charging and discharging.

Sizing a Pipe-Line Reactor. The term, pipe-line reactor, is used to designate any flow system in which: (a) reactants are fed at one end of a reactor having a large effective length-to-diameter ratio and react under conditions of varying composition (changing reaction environment) along the length of the reactor, and (b) products are withdrawn at the opposite end. In such equipment the reaction proceeds kinetically in the same way as in a batch kettle.

The volume of the pipe line for reaction only is calculated in the same manner as for a batch kettle.

Sizing a Constant-Composition Kettle. In a constant-composition kettle, raw materials are added and products are withdrawn continuously. The agitation is intense enough, in relation to the throughput, so that the composition throughout is essentially that of the effluent.

‡ If it is not feasible to maintain essentially isothermal conditions a stepwise method of calculation is employed in which the over-all reactor is treated as a series of incremental, isothermal units, each incremental unit being at a different temperature level. Data on fractional conversion at one temperature are converted to other levels by the Arrhenius equation.

Unlike a batch kettle where the rate of reaction varies from a maximum at the start of the cycle to a minimum at the end, the rate in a constant-composition kettle remains constant. For a given conversion the rate in a constant-composition kettle is equal to the minimum that prevails at the end of a batch cycle, and is equal to the slope of the tangent to the curve (Fig. 3) at the discharge conversion level. Hence the average rate is less than in a batch kettle and, for a given conversion, the continuous unit will be larger in proportion to the ratio of the average reaction rates.

To size a constant-composition kettle for this example, draw the line CB tangent to the curve at point A (Fig. 3), corresponding to the desired effluent, a conversion of 98%, and determine its slope. The slope of line CB gives the rate of reaction in a single, constant-composition kettle. Similarly, the slope of OA is the rate of reaction in a batch kettle. Hence, the ratio of these slopes is used to find the reaction time in a continuous kettle from the plot (Fig. 3) which gives the time for batch reaction.

Hold-time (constant-composition kettle) $= 77.5\left(\frac{2.5}{0.2}\right) = 970$ min.

Kettle volume (for reaction only) =

$$\frac{970}{60}(1000)(0.12) = 1940 \text{ gal.}$$

Sizing Three, Equal-Volume Cascade Kettles. In place of a single, constant-composition kettle, a series of cascade kettles is frequently used to reduce the total size (volume). The economic optimum number of kettles is a function of the specific problem, but in practice the number is rarely (if ever) greater than three. Each kettle of the series is a constant-composition unit. Thus, a large part of the total reaction may be carried out at higher driving forces than the minimum corresponding to the effluent from the last kettle in the series. The rate in each kettle of the series is constant and equals the slope of the tangent to the curve (Fig. 3) at a point corresponding to the conversion level in the unit.

A graphical method is used for determining the hold-time of each unit. The hold-time is first estimated and a graphical check is then made to see whether this estimate is correct. Usually two or three trials are required but these can be made in less than five minutes. Applying the procedure in this example:

1. Draw the line CB tangent to the curve (Fig. 3) at point A (98% conversion). The slope of the line is the rate of reaction in the last kettle of the series.

2. Estimate the time required for reaction in each kettle, say 55 min., and mark off a distance from A to C on line BC such that the horizontal projection of this distance corresponds to a 55-min. interval on the time scale.

3. Project point C horizontally back to the curve (point D) and repeat the procedure to points E, F, G, and O. Since G projects back to the origin, the estimate of 55 min. is correct. If G falls above the origin, the estimate is too low and vice versa, if below. Slopes of the tangents GF and ED are, respectively, the rates of reactions in the first and second kettles.

Volume of each kettle (for reaction only) $= \frac{55}{60}(1000)(0.12) = 110$ gal.

Notation

C_{A_0}, C_{B_0} = initial concentration of reactant A or B, moles/unit volume

C_A, C_B = concentration of reactant A or B at any time, θ, during course of reaction, moles/unit volume

f = fraction of equilibrium conversion of a reactant to product at any time, θ; f = (moles reacted)/(total moles reacted at equilibrium). Where two reactants are present, f refers to the constituent present in lower concentration.

k = reaction rate constant, consistent dimensions for different rate equations

N_R = number of reaction units, a dimensionless group of form $N_R = k\theta C_{A_0}^{m-1}$, where m = apparent order of reaction

${}_0N_R$ = number of reaction units, apparent zero-order reaction

${}_1N_R$ = number of reaction units, apparent first-order reaction

${}_2N_R$ = number of reaction units, apparent second-order reaction

r = rate of reaction, moles reacted/(unit vol.) (unit time)

θ = time of reaction, consistent units

A = a constant, dimensions dependent on reaction order

E/R = experimental constant relating effect of temperature to rate of reaction

T = absolute temperature

(*Presented at Minneapolis (Minn.) Meeting.*)

CONSTRUCTION OF ACID RECOVERY UNITS

INDOORS OR OUTDOORS?

H. S. KEMP, L. T. MULLEN, and A. P. GUESS

Du Pont Company, Inc., Wilmington, Delaware

This and the succeeding article in this number continue the series on indoor vs. outdoor construction begun in June issue with Chemical Plant Operations and The Weather. The series will conclude next month with Comparative Costs of Chemical Plants Designed for Indoor vs. Outdoor Construction.

WHEN increase in capacity for reconcentration of acetic acid by extraction with diethyl ether was required at one of the Du Pont plants, a study was undertaken to select conditions which would give the most economical installation and minimize the inherent safety hazards involved in the operation. Confronting the designers was the question: should the acid recovery units be installed indoors, as previously, or outdoors?

Sufficient company experience and know-how seemed to be available for the basic design of both the indoor and the outdoor units. Of prime importance for both types of units were the precautions that have to be taken in the handling of ether, the principal hazards of which are fire, explosion, and intoxication by inhalation of the vapors. Of equal importance for the outdoor units were the added precautions to prevent freeze-ups of glacial acetic acid when handled in equipment and piping exposed to the weather. By visits to other Du Pont Co. plants, the techniques of successfully handling glacial acetic acid in equipment and piping exposed to the weather were learned.

So that the final choice between indoor and outdoor construction of the acid-recovery units could be easily made, preliminary studies were undertaken of the following items for the two schemes under consideration:

1. Order-of-magnitude investment cost
2. Order-of-magnitude operating and maintenance cost
3. Safety features
4. Process operation
   a. Control
   b. Losses of acid and solvent

Essentially the Brewster process (*1*) for the recovery and concentration of the acetic acid is used, in which extraction of the acid with ether is followed by distillation steps. Towers are provided to recover the ether from the raffinate, and to concentrate the acetic acid in the acetic acid-water solution left after the ether has been removed from the extract. Of course, the necessary calandrias, condensers, and auxiliary equipment are provided.

The first item considered in both indoor and outdoor construction was investment cost. The basic assumption is made that the steel structure is to be the same for both indoor and outdoor construction. This is due to the fact that the towers, made in sections of thin-walled copper, are not capable of supporting the large, complicated piping arrangements along with the diaphragm valves, by-passes, and manifolds. In addition, ether reflux is not pumped but flows by gravity, and therefore support for the ether condenser is required. It is evident that substantial savings can be realized for the outdoor units in the following manner:

1. Brick walls can be eliminated.
2. Concrete foundations for the brick walls can be eliminated.
3. Lighting equipment can be reduced, e.g., by use of flood lights.
4. Building heating can be eliminated or at least reduced. (The savings from eliminating the building heating are offset to some extent by increased equipment and piping insulation.)
5. Ventilation equipment can be substantially reduced.

These various factors, associated with capital investment, indicate that the outdoor type of construction is less costly.

As to operating cost and maintenance, the performance of the process equipment should be reasonably independent of the type of building construction, provided the outdoor units are well insulated. Advantages of outdoor construction over indoor then are:

1. Reduced lighting load
2. Reduced heating load
3. Reduced ventilating load
4. Increased accessibility of the equipment
5. Concentration of the operators on one floor where they can be protected

Disadvantages of outdoor construction over indoor construction are:

1. More frequent painting of the structural steel and equipment owing to exposure to the elements
2. In severe weather, equipment maintenance is more difficult for the workers

The operating cost and maintenance factors were found to be in favor of the outdoor type of construction.

With an outdoor type the following safety features could be realized:

1. Fumes of ether or acetic acid could escape right to the atmosphere.
2. Operators could be housed in a protected central control room.
3. Protected areas for operators, in case they had to be near the equipment away from the control room, could be provided.

Of course, safety features 2 and 3 could be provided in the indoor type of construction, but not feature 1, which is the important one.

The operation of the process is essentially the same whether the equipment is indoors or outdoors. Control of the process for the outdoor type of construction must be made from a central protected control room. The developments in automatic control equipment have made it entirely feasible to house all the essential recording, indicating, and controlling instruments in a central protected location. From this central point the operator can tell at a glance what is happening at any point in the process, and can make the required adjustments. Where automatic control is not available, the operator has to go to the process equipment and take readings or adjust valves manually. With such constant checking by the operators, it was almost a necessity that the process equipment be installed indoors, with subsequent high operator exposure time near the equipment.

In the indoor type of construction, vapor losses at a faulty flange or packed joint usually can be readily detected. There was no experience available to indicate how difficult it would be to detect small leaks if the process equipment was installed outdoors, but it was felt that the problem could be handled.

Design and Construction

As a result of the preliminary study involving the comparison between indoor and outdoor construction as to capital investment, operating and maintenance cost, safety features, and process operation, it was decided to proceed with the design of outdoor units, since all the cost figures, safety features, and ease of process operation were in favor of the outdoor units. Accordingly, design was started and eventually led to construction and successful operation of the outdoor units.

A review of some of the character-

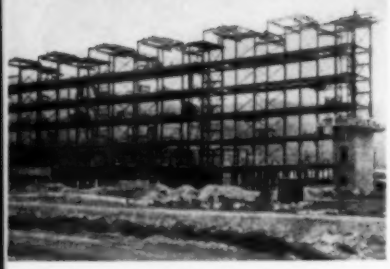


Fig. 1. Recovery units in course of construction.

TABLE 1.—SOME PHYSICAL PROPERTIES OF MATERIALS BEING HANDLED (2)

	Diethyl Ether	Acetic Acid
Molecular weight	74.12	60.05
Melting point, °C.	—116.3	16.7
Boiling point, °C.	34.6	118.1
Minimum ignition T_i, °C.	193	550
Flash point, °C.	—45	42
Lower limit of inflammability, % by volume	1.85	4.05
Specific gravity, 25/4	0.708	1.044
Miscibility with water	partial	complete

istics of the materials being handled is of interest. In Table 1 are shown some selected values of physical properties which were of importance in the design. In this table special note should be taken of the low flash point (—45° C.) for ether and the high freezing point of acetic acid (16.7° C.).

The units as finally designed and constructed in an isolated part of the plant consist of an open steel supporting structure into which are placed the various pieces of process equipment. Photographs of the construction as it progressed are shown in Figures 1 and 2. To meet the capacity requirements, three identical units were designed and constructed. The control rooms, one for each of the three units, are located at ground level. These control rooms are air-conditioned and fireproof. In the control room the operator gains complete knowledge of what is going on in the process equipment from the instruments on the panel board. A photograph of a portion of the panel board assembly is shown in Figure 3.

In case of a fire the two end stairwells, which are enclosed with brick walls, offer a place of safe evacuation for the operators. Normally, except in cases of routine inspection or repairs, no personnel are on the upper levels.

Special consideration was given to the proper sloping of all service and process pipe lines, since it is imperative that all lines be designed for complete drainage in case of extended shutdown during winter periods. Proper insulation and steam-tracing for all pipe lines and equipment are of major importance, especially when handling a material like acetic acid, whose melting point is high (16.7° C.).

Ether tanks in the tank farm were provided with water cooling to keep the temperature of the ether low during the summer. A special water tank was installed so that in case of a failure in the main water lines, sufficient water would be available for the condensers while the units were being shut down. Thus, not only could ether losses be minimized, but also a serious fire and explosion hazard from ether vapor escaping from condenser vents would be eliminated.

All wiring, electrical apparatus, and equipment such as lights, switches, panel boards, receptacles, attachment plugs, telephones, and flashlights are of the explosion-proof type. All lines and towers handling ether are grounded to avoid spark ignition, since ether flowing through a line generates static electricity. Sufficient storage capacity is provided to handle the contents of all process equipment. The storage tanks are all equipped with flame arresters.

Operation of the new outdoor units has been successful. Losses of ether and acid have been kept to a low and acceptable figure. Steam and water consumption have also been acceptable. Originally, it was felt that outdoor construction would make the detection of leaks quite difficult. Such, however, has not been the case.

No means of preventing freeze-ups, other than steam-tracing of the lines and insulation, has been used: electrical heating of the lines has not been tried. Steam-tracing and insulation have not been 100% effective in preventing freeze-ups, but when one does occur the judicious use of a steam hose has offered an effective and immediate solution.

The outdoor type of construction has been found to lend itself admirably to the handling and servicing of equipment. At the plant, where a tractor-type crane is available, it is a simple matter to approach the unit and remove equipment. Heat exchangers are lifted out of position and taken to the shops for retubing. Thus the necessity for transporting tools to the site and working in cramped quarters is avoided. The necessity for rigging up chain hoists, shoring up structural beams and the like is also avoided. Furthermore complete tower sections or complete piping manifold assemblies including control valves can be removed readily and easily for repair or replacement.

Conclusions

In the final analysis, operating department personnel claim that if they were faced with the problem today that faced them several years back, namely, indoor or outdoor units, they would not hesitate in the least to go to the outdoor type of construction.

Acknowledgment

The authors wish to express their appreciation to W. F. Cooke and C. Pyle for helpful assistance and criticisms in the preparation of this paper.

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(*Presented at Minneapolis (Minn.) Meeting.*)

Fig. 2. Recovery units at later stage of construction.

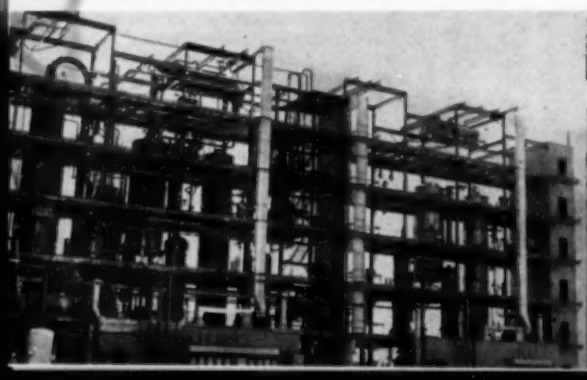
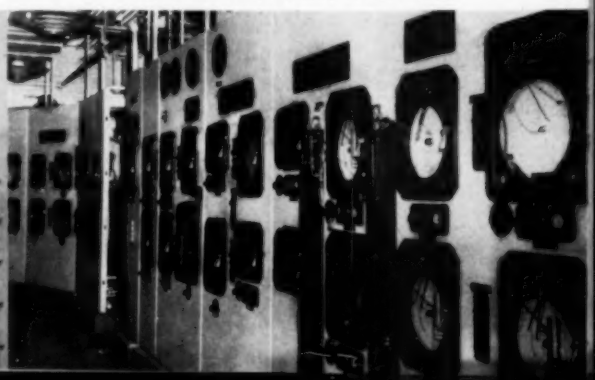


Fig. 3. Panel boards in a control room.



SAFETY AND OUTDOOR CONSTRUCTION

HOMER KIEWEG

Commercial Solvents Corporation
Terre Haute, Indiana

The principal cause of disaster perhaps in the operation of an organic chemical plant handling materials which are flammable but not explosive in nature, is explosion caused by the confinement of vapors in a restricted space. Concentrations of vapors in the explosive ranges are likely to occur due to some failure, either of equipment or operation. Prevention of explosive mixtures in confined spaces under these conditions is oftentimes impossible, and hence, even with the best of safety precautions, danger of explosion exists.

When processing equipment can be located in the open, natural air currents coupled with lack of confinement tend to dissipate the vapors quickly, reducing the concentrations to a point where they are harmless. In the event of ignition, lack of confinement can be of considerable benefit in dissipating the forces.

Use of control centers for operation reduces the frequency and severity of exposure to operating personnel, and hence the probability of human injury. Such an arrangement is possible due to the advancement in design of remote control and instrumentation.

Other problems in connection with design operation and maintenance are discussed. In weighing the economics of indoor vs. outdoor construction, consideration must be given to certain factors other than original capital investment and operating costs.

ONE of the first considerations in the basic design of a chemical plant has to do with its safe and continued operation. In the case of the average organic chemical plant this is particularly true, for in many such plants the materials being handled are flammable, toxic, or otherwise hazardous.

Of course, certain materials in themselves are explosive in nature, or can be made so by undesirable operating conditions or side reactions. They require special treatment outside the scope of this discussion. Nor is it intended to cover such recognized items of safety as the guarding of moving parts, grounding of electric systems, and handling of static electricity, which have been the subject of considerable work and discussion.

Over the course of a number of years, developments have made it possible to design plants with a minimum of housing. Regardless of the motivation for such design, it has made possible the practical elimination of certain major hazards long recognized, but which have not lent themselves to satisfactory handling.

Danger of Fire

Perhaps one of the major concerns of the designer of an organic chemical plant with respect to safety is the possibility of a disastrous fire or explosion. In the handling of solid materials or those of low vapor pressure, these problems may be minor. Fireproof construction, insulation of structural members, and adequate automatic sprinkler systems or special automatic fire-fighting equipment may give suitable and sufficient protection. But unfortunately, many processes involve the handling of highly flammable materials either readily vaporizable or with considerable vapor pressure under the conditions of operation. Failure of a piece of equipment, or a mistake in operating procedure can often result in the release of material and consequent building up of explosive mixtures in the confines of a building. Even slight leaks in the handling of toxic materials may lead to concentrations hazardous to health, despite any system of ventilation.

With indoor construction, ventilation is, of course, the obvious means for trying to eliminate these hazards. Generally, ventilating systems can be designed to take care of normal leakage or escape of vapors and fumes, but in a practical sense it is usually impossible to provide sufficient ventilation to take care of emergency conditions unless the quantity of materials being handled is small. For example, it is entirely feasible to set up a ventilating system to take care of the leakage encountered in a compressor room where gas containing sizable percentages of carbon monoxide is being compressed, even allowing for excessive leakage at the glands, but almost impossible to provide for such a contingency as the failure of a line, or cylinder, where the entire flow can escape. In most cases the physical size of the ventilating equipment, the initial capital investment, and the operating costs for heat and power will rule against an attempt at sufficient ventilation to handle such emergencies. Often ductwork itself can become a hazard due to an accumulation of lint and oil-like materials within it. This is particularly true of exhaust ducts.

Fig. 1. Pilot plant designed originally with open strip for natural ventilation. Note spandrel wall added during first cold season.

It is easy to see why the disgruntled designer should want to turn to an entirely different system of design to eliminate such a complexity of problems. Since the confining walls of a building are the major cause of the problem, why not try to eliminate them? The method has other possible merits in its favor. What are its merits from the point of view of safety? Foremost, of course, is the safety resulting from lack of confinement by a building. Furthermore, it is usually easier to space the equipment at greater distances out-of-doors without greatly increasing the cost of the installation. This in itself is highly desirable, as it reduces the exposure to damage resulting from failure of any one piece of equipment. It also makes certain phases of maintenance easier and safer. By proper arrangement a single unit can be taken out of service for repairs without the precautionary requirement of having everything shut

down and in a safe nonoperating condition as may be advisable within a building. Removal of units to maintenance shops for repair is usually easier with an outdoor installation. Often either permanent cranes or mobile hoisting equipment can be used advantageously to remove entire units.

The outdoor plant should be arranged to provide shelter and good working facilities for the personnel handling the installation. Remote control, or automatic control, is almost a necessity for such a plant, and it has only been with the development of these types of control mechanisms that it has been possible to design outdoor plants where the operator is properly sheltered. Such an arrangement reduces the exposure of the operator both to the material with which he is working, and the hazards of the process itself, compared with working in the building in which the machinery is housed. Proper spacing plus judicious installation of escapeways from elevated working areas affords excellent operator protection in the event of disaster to a plant.

Problems on Outdoor Operation

Outdoor operation of chemical operations poses certain new problems. Experience has indicated that in order to be successful, a plant built out-of-doors must:

1. Provide suitable working quarters for the operators while performing their duties
2. Allow for maintenance under suitable working conditions
3. Be designed to operate satisfactorily under the weather conditions which will prevail

As previously mentioned, it has only been with the development of control equipment that chemical processes could generally be adapted to outdoor plants. Early attempts to eliminate housing either failed to take the operator into account or at best developed into a compromise type of plant. As such, these units could hardly be considered successful.

Figure 1 is a view of such a plant. This unit was a small pilot plant handling highly flammable, and highly corrosive materials under pressure at elevated temperatures. In addition, the toxicity was unknown because of the newness of the compounds, but it was suspected that they might have a slow cumulative effect on the health of a person repeatedly exposed. Even if suitable instrumentation had been available at the time, the number of process changes that could be expected in a plant of this nature would have ruled out complete instrumentation due to the initial cost, and the time required for engineering, procurement and assembly. For these reasons it was decided to sacrifice operators' comfort and shelter the bulk of the equipment in a building which had an open band for three feet above the floor line. Before the first winter was finished, operator discomfort forced the construction of a spandrel wall as illustrated.

The operator who frequently finds it necessary to don additional clothes in order to leave inside quarters to make changes in the process he is controlling, or who finds himself exposed to rain, sleet or snow when he has to climb an exposed stairway in a structural steel tower, soon decides that the more intangible points of safety are not equal to the evident exposure and risks of his job. It becomes necessary to extend his vision and lengthen his arms in order that he can remain in his quarters and still operate his outdoor equipment.

While compressors, and other complicated pieces of machinery can conceivably be designed to function satisfactorily out-of-doors, maintenance requirements will probably dictate an indoor location. Unscheduled shutdowns may occur in any weather. The problems arising from the complexity and mechanical accuracies of the machine itself may warrant an indoor location. For example, a reciprocating shaft is more likely to wear at a gland if exposed in a dusty, outdoor location.

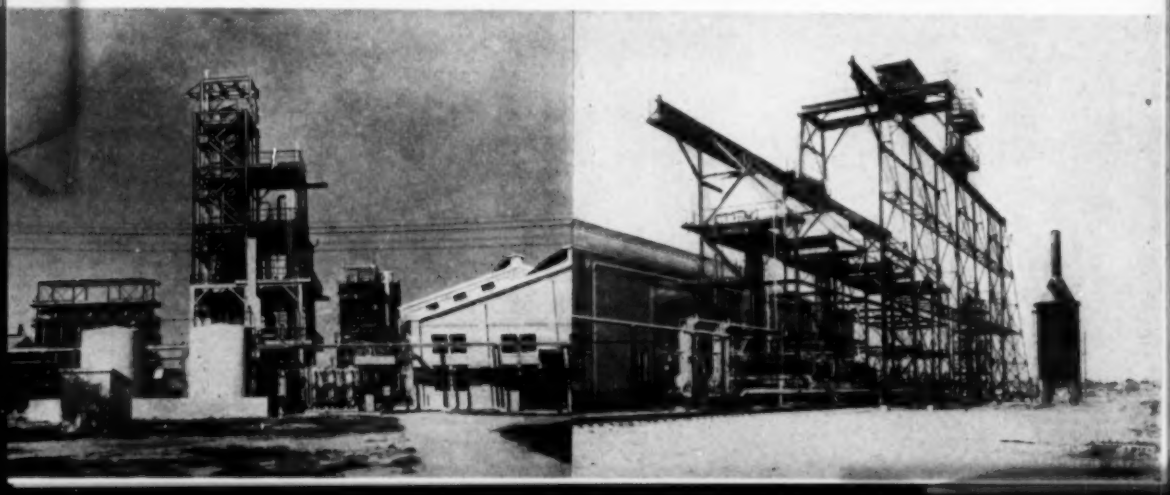
Figures 2 and 3 are views of a plant that illustrate this point. This plant, for the production of methanol, has most of the equipment out-of-doors. However, the larger building houses the compressors, circulators, and certain control equipment. The smaller one houses control equipment and minor laboratory facilities for product checking.

Another example where maintenance might govern is a tubular evaporator, on a process where frequent manual cleaning of the tubes is necessary. Operation of evaporators out-of-doors is perfectly feasible, but under the above circumstances it becomes necessary to enclose the area where cleaning is to be done.

Certain of the unit operations lend themselves readily to outdoor installations, and a plant can be designed for them that will operate under the prevailing weather conditions. Those which require manual operating labor at regular and frequent intervals are difficult to operate unhoused. A plate-and-frame filter, for example, is hardly suitable for general outdoor use. Usually there is little that can be done under these circumstances other than to place these particular operations inside. Sometimes choice of equipment, or modifications in the process may enable the engineer to eliminate housing a hazardous operation. If a process lends itself to continuous operation rather than batch, it usually becomes easier to design it for outdoor locations. This is not meant to imply that batch operations cannot be handled out-of-doors.

Figure 4 illustrates a small plant, in which complex batch distillations are handled successfully in an outside plant. The small building at the foot of the structural tower houses all controls. In

At right (Fig. 2): Outdoor location of high pressure converters. At left (Fig. 3): Methanol distillation tower.



this particular case, however, most of the lines were brought to the control house. Rotameters were mounted just outside a row of windows and valve handles extended into the building.

Advantages of Remote Control

Use of remote control is usually far superior to trying to route lines through the control room for operation. In the first place the piping usually is far too complicated and bulky for this treatment. Further, leaks occurring at packing glands, and the possibility of breaks in sight glasses, rotameters and similar equipment undo the advantages which have been gained in the outdoor installation. Transmitting pressure-indicating or -recording instruments are to be preferred over piping into the control room to a pressure gage, both from the practical as well as the safety point of view. Even an unsealed conduit housing thermocouple lines may be dangerous, since a leak occurring in a thermocouple well can make the conduit a direct pipeline for hazardous vapors. Every precaution should be taken to see that no pathway exists to bring dangerous vapors or liquids into the control room.

As remote reading instruments were improved and remote actuated valves and other devices were developed, engineering an outdoor job has become easier. Standardization, while not complete, is now sufficient so that the instrument engineer is able to build up his system from parts available from many manufacturers. Reliability has improved so that it is now possible to obtain satisfactory performance regardless of the weather conditions with maintenance comparable to that expected on any type of equipment. It should be mentioned that on air-operated systems it is vital to supply clean dry air, with the moisture content reduced below the dew point to be encountered in the coldest weather.

While reasons of safety may be one of the impelling reasons for consideration of outdoor construction, other reasons may be equally important. Safety might not dictate the outdoor location of boilers. Figure 5 is a view of such a plant built during the war when materials of construction were scarce. Turbogenerators, air compressors, switch gear and operating controls are in the building behind the boilers. All other equipment including water-treating, deaerating heaters and the like are out-of-doors. In spite of the fact that considerations of safety had little bearing in this case, some safety features may well be considered superior to those in a conventional powerhouse. No fuel is piped through the building, eliminating hazard from a broken line. Except when inspecting a boiler, the operator is protected by the building itself. Other minor points could be mentioned, which make the outdoor design of such a plant an attractive choice from the point of view of safety.

Summary and Conclusions

In the construction of a chemical plant, outdoor construction lends itself to design which reduces the possibility of some of the hazards difficult to cope with if housed construction is used. It greatly reduces the possibility of accumulation of flammable vapors in concentrations sufficiently high to be in the explosive range. It reduces the exposure of allied equipment in the event of failure of one piece of apparatus. It reduces the operator's exposure to fumes which may be toxic. It lessens the possible physical danger to the operator in the event of an accident involving fire. To cope successfully with these problems when designing an enclosed plant is difficult at best and it is usually impossible to achieve comparable degrees of safety.

Outdoor construction, where a process lends itself to this type of design, can be arranged for comfortable operation, and close process control, and in so doing provide even more protection for the operator by reducing the need for his remaining in the immediate vicinity of the operating equipment.

In order to design an outdoor plant successfully, the process must be able to meet certain requirements. Some operations or equipment do not lend themselves to this treatment. While the only alternative may be to house sections of a process, consideration should be given to alternate methods of processing.

The development of remote reading instruments, remote operating devices and automatic control has extended the possibility of outdoor plants. Judicious choice of instruments and careful installation are sometimes important to eliminate dangers in the control house.

Because of economic or other reasons, outdoor installations may be advisable and safety may not be a serious consideration. Even under these conditions, often it will be found that the result is a safer plant.

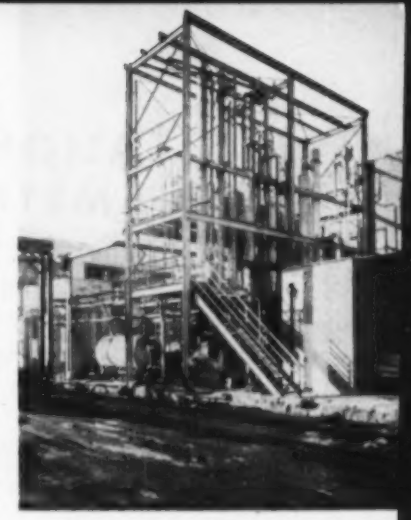


Fig. 4. Outdoor plant with batch distillation systems.

Acknowledgment

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(*Presented at Minneapolis (Minn.) Meeting.*)

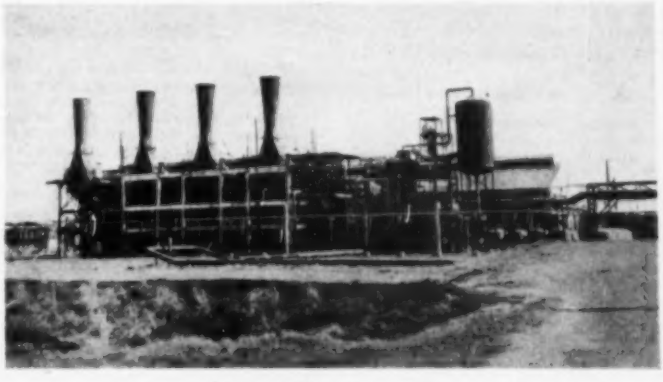

Fig. 5. Outdoor boiler plant.
Controls and turbogenerators in building in background.

DISTILLATION IN 2, 4 AND 5.75 INCH DIAMETER PACKED COLUMNS

A. C. HEINLEIN †, R. E. MANNING, and M. R. CANNON

Pennsylvania State College, State College, Pennsylvania

The efficiencies of two types of protruded metal packings and 100 mesh, ¼-in. McMahon packing were determined in 2, 4, and 5.75-in. diameter columns. Extensive data on efficiency, holdup and pressure drop over the complete operating range were obtained. Protruded metal packing (hole size B) was found to be the most efficient in all three columns, showing the lowest H.E.T.P. and the lowest pressure drop per plate. In type 316 stainless steel it is approximately 50% of the cost of McMahon packing per theoretical plate for a column 4 in. I.D. In a 6-in. diameter column the economic advantage is even greater.

The effect of physical properties of liquid on holdup and flooding velocity in packed columns was determined by distilling the following pure compounds: normal heptane, methylcyclohexane, benzene and ethylene dichloride. It was found that, at any given vapor velocity, the holdup was largest for the liquid of lowest density. Flooding velocity was largest for the most dense liquid. For example, a packing that flooded at a superficial vapor velocity of 1.55 ft./sec. with normal heptane flooded at 2.2 ft./sec. with ethylene dichloride.

Liquid velocities down through the packing, at any given vapor velocity, varied with test mixture and packing. For example at superficial vapor velocity of 1.2 ft./sec. the liquid velocity for the normal heptane-methylcyclohexane test mixture was .034 ft./sec., whereas for the benzene-ethylene dichloride system it was .027 ft./sec.

THIS report covers the efficiency of two protruded packings and ¼-in., 100-mesh McMahon packing in columns 2, 4 and 5.75-in I.D. In addition data are presented which show that holdup, flooding velocity and liquid velocity for a given packing are quite different for different liquids. Cost data for the various packings are given.

Test Equipment

Columns. The 2- and 4-in. diameter columns have been described (*4*). The 5.75-in. I.D. column was constructed in a manner similar to the 4-in. column. All stills were steam-heated with automatic control of boil-up rate by means of an electric solenoid valve in the steam line. The control instrument was a water manometer containing a few drops of sulfuric acid connected to the base of the column and containing one fixed and one movable electrode. These electrodes were part of a circuit through the coil of a relay which operated the steam valve. The movable electrode was set to the desired pressure and the steam flow was then automatically controlled to maintain this pressure. The resulting control of boil-up rate was excellent—variations did not exceed 3%.

Distributor Plates. Construction of the distributor plates has been described (*4*). For the 2-in. diameter column five liquid return tubes extended 1 in. from the base of the plate. One of these was in the center and the other four on a 0.56-in. radius. The 10-32 screws threaded into the bottom of these tubes contained orifices 0.042 in. in diameter. Four vapor risers ½-in. I.D. extended 2 in. above the plate.

The distributor plate for the 4-in. column contained one liquid return tube in the center and six on a 1-in. radius with orifice diameters of .073 in. Ten ½-in. I.D. vapor risers were used, four on a ½-in. radius and six on a 1½-in. radius.

The distributor plate for the 5.75-in. I.D. column was constructed similarly with one liquid return tube in the center, six on a ¾-in. radius and twelve on a 1⅝-in. radius. Twelve vapor risers were on 1⅛-in. radius and twelve on a 1⅝-in. radius. The orifice diameter in the machine screws was .0595 in.

Test Liquids

The normal heptane and methylcyclohexane were specially prepared by the manufacturers for highest possible purity. They were each redistilled in an 80-plate packed column at a reflux ratio of 60 and found to be pure. The benzene was found to contain some impurities to the possible extent of 3% and the ethylene dichloride to the possible extent of 1%.

Analyses of the *n*-heptane-methylcyclohexane mixture were made by a five-place Valentine refractometer and a four-place Abbe type at 20.0° C. using the data of Bromiley and Quiggle (*3*). The pure methylcyclohexane had an $n_D^{20} = 1.4232$ and *n*-heptane an $n_D^{20} = 1.3877$.

Theoretical plates at total reflux were calculated using an $\alpha = 1.070$ recommended by Beatty and Calingaert (*1*) and used by Fenske (*5*).

Experimental Procedure. The still was charged with test liquid and the condenser water and steam valves opened. Because of excellent heat-transfer coefficients the largest column was brought to the flood point within 20 min. and the smaller diameter columns in less time. The electrode in the control manometer was then set to the pressure drop corresponding to the desired operating rate and the flooded packing settled to steady operation within 10 to 15 min.

Dynamic holdup was determined by isolating the column from the still and draining the packing for 1 hr. The static holdup remaining in the packing was measured in the manner previously described (*4*) and added to the dynamic.

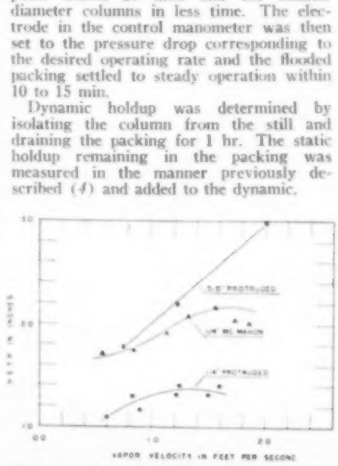

Fig. 1. Packing efficiencies in a 2-in. diameter column with a packed height of 118 in.

Experimental data (Tables 4-6) are on file (Document 3181) with the American Documentation Institute, 1719 N Street, N.W., Washington, D. C. Data obtainable by remitting $1.00 for a microfilm and $1.35 for photocopy.

† Present address: Merck & Company, Rahway, N. J.

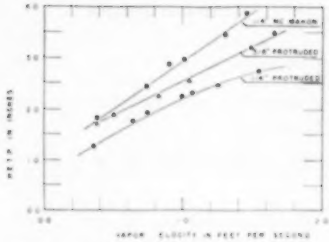

Fig. 2. Packing efficiencies in a 4-in. diameter column with a packed height of 115 in.

Pressure drops were read by both water and mercury manometers.

When theoretical plates were to be determined, the refractive index of the distillate and reflux from the base of the column was measured every half hour. Plates were calculated after these readings showed no change.

Liquid velocities were measured only in the 2-in. column. This was done by making a concentrated solution of distillate and nonvolatile red dye and injecting it into

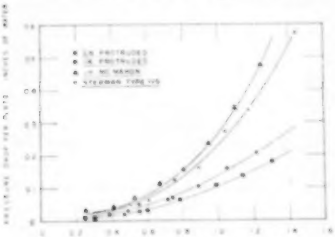

Fig. 4. Pressure drop per theoretical plate for packings in a 5.75-in. diameter column with a packed height of 73 in.

the top of the packing and starting a stop watch at the same instant. In order to detect its appearance at the base of the column it was necessary to have the liquid drop into the glass reflux measuring tube while it was maintained approximately half full of liquid. The lineal liquid velocity reported is the length of the packed section divided by the flow time of the dye.

Performance of Packings

Figures 1, 2 and 3 show the efficiencies of McMahon and protruded metal

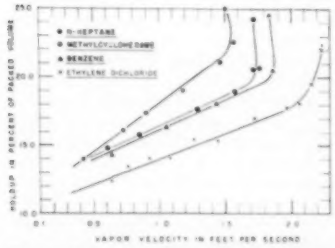

Fig. 6. Effect of liquid properties on the holdup and flooding velocity. Column diameter 2 in., packed height 120 in., protruded nickel packing.

packings in columns 2, 4 and 5.75-in. I.D. Note that ¼-in. protruded stainless steel is the most efficient packing by a large factor in the 2-in. column. However, in the 4-in. diameter column there is only a slight difference between the ¼-in. and ⅜-in. protruded packings and in the 5.75-in. diameter column there is practically no difference.

Katz (*8*) has measured the efficiency of these same packings in a 1-in. diameter column with packed height of 41 in. Over the range of 1 to 3.0 l. of liquid reflux/hr. he found the *H.E.T.P.* of ¼-in. protruded stainless steel to vary from 1.1 to 1.4 in.; ¼-in. McMahon, 1.3 to 1.6 in.; ⅜-in. protruded, 1.3 to 1.6 in.

The large effect of hole size and consequently burr size was surprising. Hole size *A* (.27 × .37 mm.) is less efficient and has a higher pressure drop per plate than hole size *B* (0.40 × .37 mm.). Consequently, it is no longer in production. A previous publication (*4*) shows hole size *A* in protruded nickel to have high efficiencies in 2- and 4-in. diameter columns.

Figure 4 shows ¾-in. protruded stainless steel to have a much lower pressure drop per plate than either Stedman or McMahon packing in a 5.75-in. diameter column. The Stedman data are those of Bragg (*2*) determined with the system benzene-ethylene dichloride. Because of the high liquid phase density of this system a much different type of graph would result if the ordinate of Figure 4 were boil-up rate in gallons of liquid per hour. For such a plot the pressure drop per theoretical plate for Stedman packing is five times that for protruded packing.

If the pressure drop per plate were plotted for the above packings in 2- and 4-in. diameter columns similar curves would be obtained.

Figure 5 shows that the holdup per plate for protruded packing is approximately half that of ¼-in. McMahon packing in a 5.75-in. column.

Effect of Liquid-Phase Properties on Packed Column Behavior

Holdup and Flood Rate. Figure 6 shows that liquid phase density has an important influence on holdup and flooding velocity. For example, the holdup for normal heptane at a vapor velocity of 1.4 ft./sec. is 140% that of ethylene dichloride. If it is assumed that in each case the same number of square feet of packing is wet, then the thickness of the liquid ethylene dichloride film would be 38% that of the liquid normal-heptane film.

Similarly, the flooding velocity of ethylene dichloride is 47% greater than that for the less dense normal-heptane.

The velocities of the liquid phase

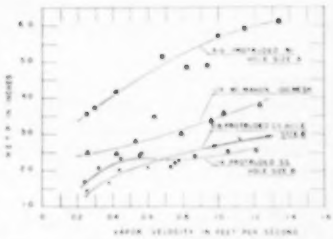

Fig. 3. Packing efficiencies in a 5.75-in. diameter column with a packed height of 73 in.

down through the packing for the test system normal-heptane-methylcyclohexane and the system benzene-ethylene dichloride are presented in Figure 7. To date no practical use has been made of such data but they may prove to be of value in later studies.

Table 1 lists the viscosities of the test liquids at their boiling points. Other things being equal it is the kinematic

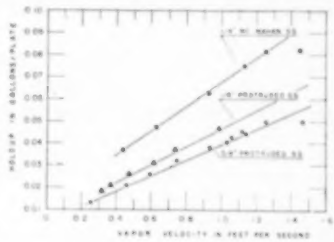

Fig. 5. Holdup for packings tested in 5.75-in. diameter column with a packed height of 73 in. Measured with the system n-heptane-methylcyclohexane.

viscosity rather than absolute viscosity that determines the thickness of a liquid film flowing under the influence of gravity. The 50 mole % mixture of the two systems differs in kinematic viscosity by only 8%. This means that viscosity should have little influence on the difference in holdup, flood rate and liquid rate data obtained for the two systems.

It is now clear that authors should

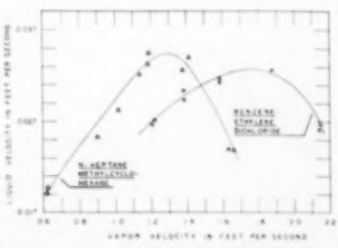

Fig. 7. Liquid velocities at total reflux through ¼-in. protruded packing in a 2-in. diameter column with a packed height of 118 in.

TABLE 1.

PROPERTIES OF TEST LIQUIDS AT THEIR BOILING POINTS

Test Liquid	Kinematic Viscosity centistokes	Absolute Viscosity centipoises	Density at B. P. gm/cc
Benzene	0.385	0.320	0.81
Ethylene dichloride	0.367	0.425	1.16
n-Heptane	0.292	0.180	0.62
Methylcyclohexane	0.379	0.265	0.70
50 mol % Benzene 50 mol % $C_2H_4Cl_2$	0.368	0.360	0.98
50 mol % n-Heptane 50 mol % Methylcyclohexane	0.341	0.222	0.65

Viscosities were measured at 20°C and 40°C; calculated at boiling point by Andrade equation.

not compare holdup and flood rate data on packings that are tested with different test mixtures. Impurities in the benzene used in this work made it impossible to compare plate values for the same packings when tested with the two different test mixtures. However, ¼-in. McMahon packing has been tested (*7*) in a 6-in. diameter column with benzene-ethylene dichloride. *H.E.T.P.*'s of 2.1 to 3.9 in. were obtained. This is in good agreement with the data reported here. While Fisher and Bowen (*6*) recommend the system benzene-ethylene dichloride the present authors prefer the system *n*-heptane-methylcyclohexane for three important reasons. These are: (a) It can be used to test more efficient columns, (b) Much data on various packings obtained with this mixture are now in the literature and (c) Both components are commercially available in a high degree of purity.

Cost Comparison of Packings

Table 2 lists physical data on the packings tested. Table 3 contains cost comparisons. Larger sizes of protruded packing are now being tested. As packing size increases the cost per cubic foot sharply decreases. In the sizes now available it is evident that protruded packing is one of the most economical.

TABLE 2.

PHYSICAL DATA ON PACKINGS

Type	Normal Size (In.)	Actual Size (In.)	Material of Construction	Number Per Cu. Ft.	Area Sq. Ft. Per Cu. Ft.	% Free Space
Protruded	1/4	.16 x .16	Type 316 Stainless Steel	800,000	576	94.0
Protruded	3/8	.24 x .24	Type 316 Stainless Steel	290,000	396	96.0
McMahon	1/4	-	Type 316 Stainless Steel; Wire Diameter .0045 in.	450,000	610* 419**	95.3* 85.3**

Protruded holes per square inch = 1024

Protruded hole size A = 0.37 x 0.27 mm.

Protruded hole size B = 0.40 x 0.37 mm.

*Assuming holes in mesh are not sealed with liquid (6)

**Assuming holes in mesh are sealed with liquid (6)

TABLE 3.

APPROXIMATE COST OF SEVERAL PACKINGS FOR A ¼ INCH DIAMETER COLUMN

Line	Type of Packing	Packing Cost Per Plate	Packing Plus Col. Per Plate
1	0.50 Inch Berl Saddles (Ceramic)	$ 1.50	$ 7.25
2	0.50 Inch McMahon Saddles (Monel)	4.20	7.25
3	0.25 Inch Berl Saddles (Ceramic)	6.00	9.00
4	0.50 Inch McMahon Saddles (Stainless Steel)	7.25	10.25
5	0.25 Inch McMahon Saddles (Brass)	9.05	11.25
6	0.25 Inch McMahon Saddles (Monel)	13.30	15.25
7	0.25 Inch McMahon Saddles (Stainless Steel)	14.00	16.25
8	0.375 Inch McMahon Saddles (Stainless Steel)	12.05	14.50
9	Stedman Triangular Pyramidal (Stainless Steel)	31.80	39.00
10	0.25 Inch Protruded Stainless Steel	6.04	7.59
11	0.375 Inch Protruded Stainless Steel	5.00	7.66

Note: Lines 1 to 9 taken directly from reference (6)

Lines 10 and 11 calculated from data in this report

Acknowledgment

The authors wish to express their appreciation to the Scientific Development Co. of State College, Pa., for donating the protruded packings for this work.

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Discussion

C. R. Bartels (E. R. Squibb & Sons, New Brunswick, N. J.): Apparently the hole size has quite an effect on the efficiency of your packing. Are you satisfied that you have the optimum size now?

R. E. Manning: No, to date we have tried only two different sizes. We intend to experiment with different sizes. Hole size *A* is .27 × .37 mm. and hole size *B* is .37 × .40 mm. One further addition on this, it was found that hole size *A* and *B* had about the same efficiencies in small diameter columns but that *B* was much better than *A* in a 6-in. diameter column. Evidently things are in fine state of balance for two different packings to have the same efficiency in a small column and much different efficiencies in a large column.

C. S. Carlson (Standard Oil Development Co., Linden, N. J.): I notice in one of your graphs you showed a 20% holdup. Am I to understand that represents 200 cc. of liquid held in the column?

R. E. Manning: Yes, for a packed volume of 1000 cc. At low vapor velocities the holdup may be only 10 or 12% of packed volume.

C. S. Carlson: In another graph you talk about large diameter columns. We had experiences with this type of packing in the smaller sizes. In normal size columns, 1 in. in diameter, we find this type effective for use under conditions such as extractive distillations with high liquid and vapor loadings.

(*Presented at Forty-second Annual Meeting, Pittsburgh, Pa.*)

MIXED HYDROCARBON SOLVENTS FOR FATTY ACIDS AND THEIR TRIGLYCERIDES†

RICHARD BOGASH‡ and A. NORMAN HIXSON

University of Pennsylvania, Philadelphia, Pennsylvania

The solubility relationships of fatty acids and their triglycerides in mixtures of methane and propane have been investigated and compared with those in pure propane. It is shown that the addition of methane effectively lowers the critical solution temperature for a given solute.

Equilibrium data are presented for the system stearic acid-propane-methane at 85.4° C. and 71.2° C. and for the system trimyristin-propane-methane at 69.6° C. and 61° C. The corresponding critical solution temperature for the propane binary solution is 91.4° C. for stearic acid and 79.4° C. for trimyristin.

A method was presented for the determination of conjugate liquid compositions from equilibrium data obtained by the synthetic method when the pressure is always in equilibrium with one or two liquid phases.

Evidence of the importance of solvent density is presented and quantitatively compared with the predictions from a generalized solubility equation of Hildebrand.

It is shown how the use of methane mixtures increases the versatility of propane as a selective solvent.

THE unusual solvent properties of propane have led to its increasing use in liquid-liquid extraction processes. It has been employed singly or in combination with other solvents in the petroleum industry for the refining of lubricating oils (*13*). More recently, investigations have shown it to be effective in the separation of fatty acids and their triglycerides (*3, 5, 8, 9*). There are at present several commercial processes using propane for the purification of the fatty acids and their triglycerides (*16*).

The usefulness of propane as a selective solvent arises fundamentally from the reverse temperature-solubility curve exhibited by some of its binary solutions. Certain pure compounds although completely miscible in propane at room temperature separate out and form a second immiscible liquid layer as the temperature is raised (*3, 5, 8, 9*). The temperature at which this reversed solubility occurs is a function of the chemical nature of the solute, its molecular weight, and the composition of the binary mixture. These binary systems uniformly show a U-shaped temperature-composition curve with a minimum point, the temperature below which all compositions are miscible. Differences between their critical solution temperatures, or lower consolute points, have proved a valuable qualitative index in estimating the possibility of separating a mixture of two compounds by propane. In addition, a useful correlation relating the critical solution temperature to the effective molecular weight of the

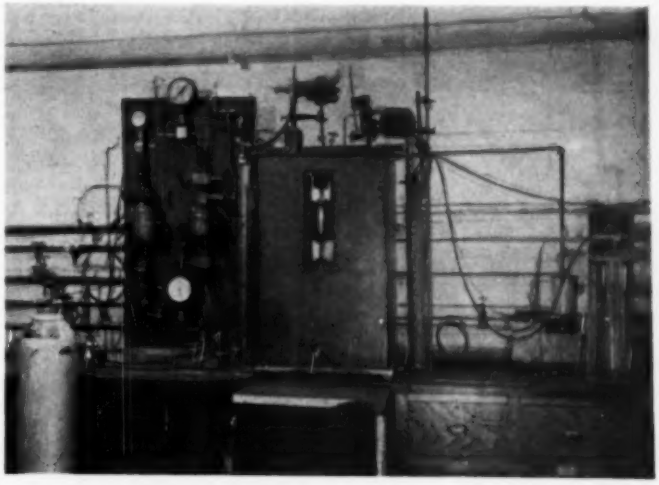

Fig. 1. Photograph of apparatus.

† Complete details of apparatus and experimental technique are available at University of Pennsylvania.

‡ Present address: Wyeth, Inc., Philadelphia, Pa.

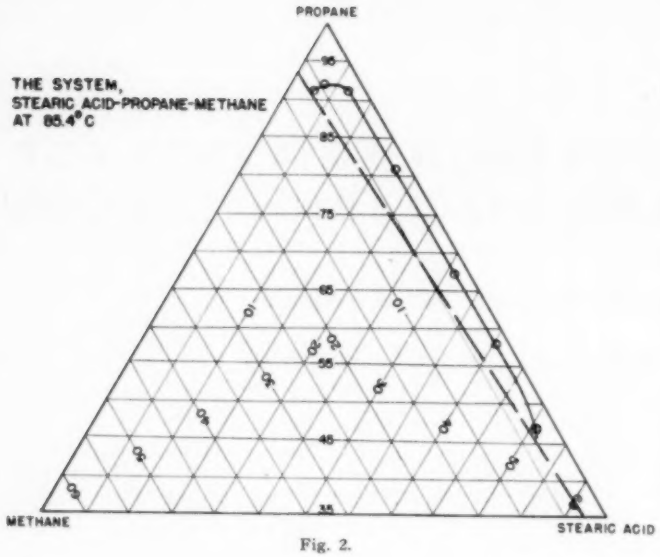

Fig. 2.

TABLE 1

PRELIMINARY INVESTIGATION OF THE SOLUBILITY OF FATTY ACIDS AND TRIGLYCERIDES IN PROPANE - METHANE MIXTURES

Solute	Phase Separation Temperature	Critical Solution Temperature of Solute and Propane	Pressure	Composition in Weight % Solute	Methane	Propane
Stearic Acid	98.3 °C.	91.4 °C.	625 lb./sq.in.	19.4	1.3	79.3
	85.5		663	23.3	1.7	75.0
Palmitic Acid	86.0	96.9	720	14.9	2.6	82.5
Trimyristin	71.3	79.4	590	14.5	2.5	83.0
Trilaurin	80.3	89.7	710	12.0	2.9	85.1

solute in a simple binary system with propane has been proposed and tested for a number of fatty acids and their esters (*3, 5*). The effect of other functional groups upon solubility and critical solution temperatures in propane has also been surveyed in studies with typical nitriles, amines, and amides (*3*).

TABLE 2

THE SYSTEM, STEARIC ACID - PROPANE - METHANE

Composition in Weight Per Cent Stearic Acid	Methane	Propane	Temperature °C.	Pressure lb./sq.in.	Composition (solute-free) Mol Per Cent Methane	Propane
3.7	3.7	92.6	85.4	620	9.9	90.1
7.7	2.1	90.2	"	662	5.8	94.2
18.3	1.8	80.2	"	652	4.9	95.1
32.4	1.2	66.4	"	632	4.7	95.3
41.8	1.4	56.8	"	708	6.3	93.7
50.6	1.8	47.6	"	620	9.5	90.5
17.1	4.0	78.9	71.2	745	12.3	87.7
29.3	3.5	68.2	"	740	12.3	87.7
34.2	2.9	62.9	"	748	11.3	88.7

TABLE A

Solute	Actual M.P. °C.	M.P. of Pure Material °C.
Trilaurin	46.0-46.5	46.5 (*1*)
Trimyristin	56.8-57.2	56.5 (*1*)
Stearic acid	69.2-69.5	69.6 (*6*)
Palmitic acid	62.5-63.0	62.9 (*6*)

This work was undertaken to investigate the potential applications of solvent mixtures of propane and other low molecular weight hydrocarbons for the separation of fatty acids or their triglycerides. As typical examples, certain ternary systems, propane-methane-fatty acid or triglyceride, were studied at selected temperatures. Furthermore, it was thought that the ternary solubility behavior of these mixtures would materially aid in explaining the phenomenon of reverse solubility in these relatively nonpolar systems.

Apparatus. Phase relationships for the ternary systems were observed in small Jerguson gauge (No. 2 OWP) equipped with an air-driven stirrer previously described (*3*). A picture of the apparatus is shown in Figure 1. A supplementary mercury displacement apparatus was used to add measured amounts of methane at any desired pressure.

General Procedure. In a ternary system at equilibrium, when three phases coexist (two liquid and one vapor) under conditions of fixed temperature and pressure, the only independent variables are the concentrations of two of the components in either liquid phase. To fix such a point on the solubility curve the third component, methane in these experiments, was added slowly (at constant temperature) to a given mixture of solute and propane until the second phase started to appear. This method, termed the synthetic method, was used throughout the investigation.

The solute was weighed and charged to the gauge. The amount of propane and methane in the liquid phase was determined indirectly. The total volume of methane charged was known, and from the compressibility data of Kvalnes and Gaddy (*12*) its weight was calculated. The total volume of propane and methane in the Jerguson gauge was measured in a gasometer at atmospheric pressure at the conclusion of the experiment making the proper correction for the vapor pressure of the sealing water. The volume of methane charged was subtracted to obtain the volume of propane. Its weight was calculated with the aid of previously published compressibility data (*2, 4*). There then remained only a small correction for the methane and propane in the vapor phase. This never amounted to more than 10% of the total charged and usually was about 5%. For this correction, the density and composition of saturated propane-methane vapor in equilibrium with its liquid at the observed pressure and temperature of the system were used (*15*). As shown before there is a negligible error in assuming the gas phase to be completely hydrocarbon (*5*). The accuracy of the calculation depends upon the effect of the solute on the

relative volatility of methane and propane. The assumption that this is negligible is reasonable since the solutes were almost nonpolar. However, even if the densities determined for saturated conditions were 20% in error, an over-all error of only 2% would result in the composition of the liquid propane-methane mixture because of the small size of the vapor space.

Materials. The propane and methane were obtained from the Matheson Co., East Rutherford, N. J. Both were specified to be at least 99% by weight pure, the impurities being other saturates. Trilaurin and trimyristin were obtained from Eastman Kodak Co. Both were recrystallized once from ethanol. Stearic acid and palmitic acid were supplied by Armour and Co. in about 90% purity, the other 10% being primarily palmitic acid in the stearic and vice versa. Each was recrystallized four times from ethanol. (*See* Table A.)

Results

Table 1 presents the data from a preliminary investigation of the solubility of fatty acids and triglycerides in propane-methane mixtures. Stearic acid, palmitic acid, trimyristin, and trilaurin were tested because complete phase diagrams had already been determined for each with propane alone. In all four cases, the ternary systems methane-propane-solute exhibited two well-defined liquid phases at temperatures well below the critical solution temperature for the corresponding binary mixture of propane-solute. The amount of methane necessary for phase separation depended upon the temperature, the nature of the solute, and the initial composition of solute in propane.

The critical solution temperature for a binary mixture of propane and stearic acid was 91.4° C. (*5*) Below this temperature there is only a single liquid phase regardless of composition. Test runs were made at 88.5° C. and 85.5° C. with methane as a third component. In the first instance, phase separation was observed at the following composition in weight per cent, methane 1.2, propane 79.3, and stearic acid 19.5; in the second (at lower temperature), methane 1.7, propane 75, and stearic acid 23.3. The results for palmitic acid, trimyristin, and trilaurin were similar to those for stearic acid and the details may be found in Table 1.

On the basis of the preliminary data, the ternary systems, methane-propane-stearic acid and methane-propane-trimyristin, were chosen for more complete study. The phase diagram for methane-propane-stearic acid at 85.4° C. is shown in Figure 2. The composition and pressures at phase separation are recorded in Table 2. The diagram is typical for a ternary mixture consisting of two pairs of completely miscible substances, methane and propane (in all possible combinations below the critical pressure), and propane and stearic acid. The third pair, stearic acid and methane (entirely hypothetical at this temperature) would be almost immiscible. In the diagram, the area within the curve represents a two-phase region while outside the curve there is complete miscibility. The curve itself is relatively flat except at its extremities where large increases in methane concentration are necessary to produce phase separations. The entire two-phase region extends only from 3 to 47% by weight solute. The dotted line across the diagram designates the composition of the propane-methane mixture (solute-free basis) having a critical temperature of 85.4° C. It should be noted that the diagram is abbreviated in order to enlarge the scale. The points labeled Stearic Acid and Methane do not represent 100% as shown.

The essential part of the diagram, covering the solute concentrations of 19 to 34% by weight, was rerun at a lower temperature, 71.2° C. (Fig. 3). Similar phase separations were again obtained, but with somewhat higher methane concentrations.

The system, trimyristin-propane-methane, was also investigated quantita-

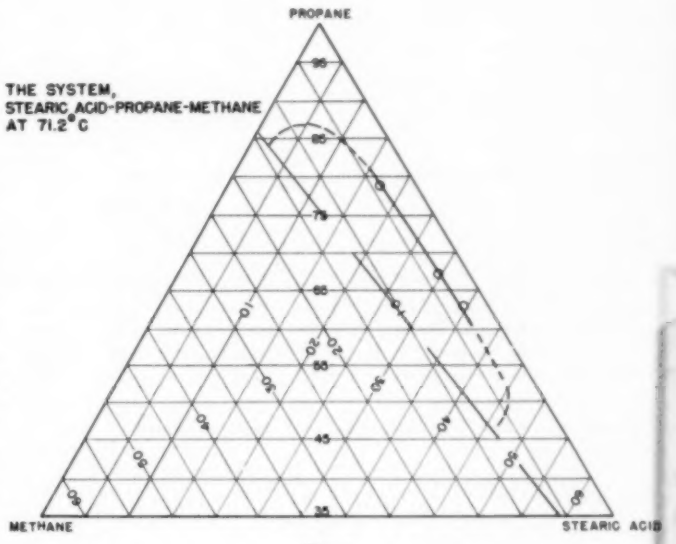

Fig. 3.

TABLE 3

THE SYSTEM, TRIMYRISTIN - PROPANE - METHANE

Composition in Weight Per Cent			Temperature °C.	Pressure lb./sq.in.	Composition (solute - free) Mol Per Cent	
Trimyristin	Methane	Propane			Methane	Propane
4.0	4.6	91.4	69.6	682	12.1	87.9
6.0	3.5	90.5	"	630	9.8	90.2
8.3	3.4	88.3	"	680	9.5	90.5
17.5	2.5	80.0	"	580	7.7	92.3
34.6	1.7	63.7	"	580	7.0	93.0
42.8	1.7	55.5	"	630	7.5	92.5
47.5	1.7	50.8	"	640	8.3	91.7
55.9	2.2	47.9	"	730	12.1	87.9
57.5	2.0	40.5	"	715	12.0	88.0
17.1	3.9	79.0	61.0	640	11.8	88.2
32.3	2.9	64.8	"	637	11.1	88.9
42.6	2.5	54.9	"	638	10.9	89.1

TABLE 4

CONJUGATE LIQUID COMPOSITIONS

Extract			Raffinate		
Weight %			Weight %		
Trimyristin	Propane	Methane	Trimyristin	Propane	Methane
51.6	46.7	1.8	5.0	91.5	3.5
45.8	52.5	1.7	7.5	89.0	3.5
42.0	56.2	1.8	10.0	87.0	3.0
39.2	59.2	1.6	12.5	84.5	3.0
37.0	61.2	1.8	15.0	82.0	3.0
34.5	63.5	2.0	17.5	80.0	2.5
33.0	65.0	2.0	20.0	77.5	2.5

TABLE 5

SOLVENT DENSITY AT INITIAL PHASE SEPARATION FOR THE PRELIMINARY RUNS

		Propane				Methane-Propane			
Solute	Wt. % Solute	Solvent Density	Pressure	Temp.	Critical Sol'n. Temp.	Solvent Density	Pressure	Temp.
Trilaurin	12.0	0.344 gm./cc.	516 lb./sq.in.	97.9°C.	97.4°C.	0.357 gm./cc.	690 lb./sq.in.	80.3°C.
Trimyristin	14.5	0.376	458	79.9	79.4	0.373	590	71.3
Stearic Acid	19.4	0.325	551	91.6	91.4	0.325	625	80.5
	23.3	0.324	553	91.4	91.4	0.324	632	80.5
Palmitic Acid	14.9	* ---	601	98.9	98.9	0.296	730	80.5

* Propane alone is above its critical temperature at the temperature of phase separation.

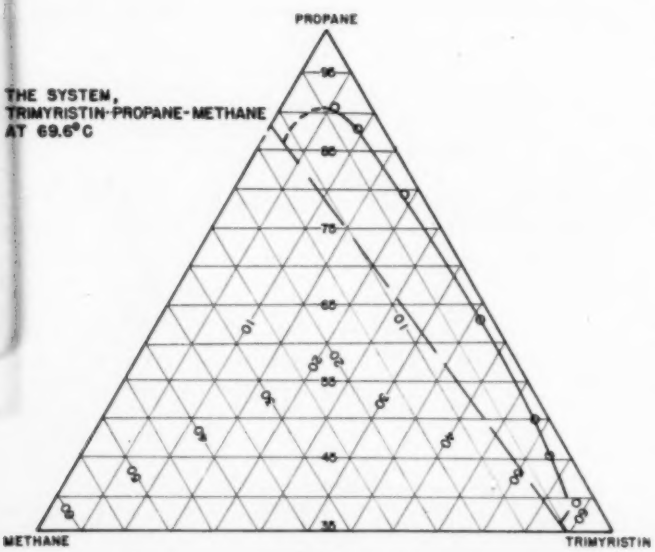

Fig. 4.

tively, and the phase diagram and table of results at 69.6° C. are found in Figure 4 and Table 3. The diagram is similar to the one for stearic acid, but less methane is needed for phase separation at comparable temperatures and compositions. The two liquid phases are obtained from about 4 to 54% by weight solute. Once again, parts of the diagram were also determined at a lower temperature, 61.0° C. This diagram and table are shown in Figure 5 and Table 3.

Conjugate Liquid Compositions. The major disadvantage of the synthetic method in the study of phase relationships is the inability to secure "tie-line" data, the compositions of the two liquid phases in equilibrium. In the systems investigated in this work, however, this information can be calculated since the total pressure was always the pressure in equilibrium with one or two liquid phases. At the point of initial phase separation the total liquid composition is the same as that of one of the phases. The pressure at this point is also the same as that in equilibrium with the conjugate layer. Since the solute in these systems (volatile hydrocarbon and high molecular weight acid or triglyceride) has always been considered to be totally in the liquid phase, a simple plot of pressure (at initial phase separation) against weight per cent solute gives a curve on which two solute concentrations linked by a line of constant pressure represent the solute compositions of conjugate layers. With the aid of the ternary phase diagram, these solute compositions can be translated into total compositions for each layer. Actually the limitation that the solute be completely in the liquid phase need not apply as long as the weight of the components in the vapor phase is negligible in comparison with the liquid phase.

The accuracy of this method in determining equilibrium concentrations depends upon the precision of the pressure measurements. This investigation used only a well-calibrated Bourdon pressure gauge since its primary purpose was not that of securing tie-line data. However, the method of calculation is interesting and does have particular value where sampling is impossible or the analysis is difficult. For this reason, its application to the data for the ternary system, methane-propane-trimyristin has been included.

The graph in Figure 6 is a plot of the pressure at initial phase separation against weight per cent trimyristin at 69.6° C. As explained previously, the solute concentrations of the two layers in equilibrium were obtained by traversing a line of constant pressure. In Figure 7, the envelope conditions for

the ternary diagram are replotted in terms of only weight per cent propane and weight per cent trimyristin. From the solute concentrations, the propane concentrations were then found. The methane concentrations were, of course, 100-weight per cent propane-weight per cent trimyristin. Table 4 lists some of the compositions of conjugate liquid layers.

Comment. In previous work with propane and fatty acids or triglycerides it was suggested that changes in the liquid density near the critical temperature of propane were responsible for the reversed solubility phenomena. In this investigation methane was chosen as the third component using the same systems so that the liquid density could be varied (diminished) at a given temperature and the subsequent solubility changes observed. Because of the similarity of methane and propane, their nonpolar character, their inability to solvate or to compound with the solute, it was thought that any changes in the solvent power of the liquid medium could be traced to density changes.

A direct measurement of the liquid density at the point of initial phase separation was not feasible in this equipment, nor was such information available for comparison with the binary systems with propane alone. Instead, the density of the solvent alone at the conditions of phase separation was chosen as a basis of comparison. For propane alone and propane-methane mixtures this information was available (*2, 4, 15*).

For the preliminary runs, the data are included in Table 5. At the same concentration of solute, the supposition was that the densities would be about the same either with propane alone or with propane-methane mixtures at the point of insolubility. The results were encouraging in that over a wide diversity of conditions at phase separation the solvent densities for propane alone and propane-methane were almost identical. This analysis was carried out for both complete ternary systems, the one with stearic acid, the other with trimyristin.

For the sake of comparison, the solvent densities at initial separation for both the ternary and binary systems were plotted on the same graph against the weight per cent solute, Figure 8 for trimyristin and Figure 9 for stearic acid. The density values from which the graphs were constructed are listed in Table 6. The curves are inverted U's showing the maximum density at which phase separation can occur. For trimyristin in liquid propane, Figure 9, curve *A*, this maximum density is 0.380 g./cc., in propane-methane mixtures at 69.6° C., curve *B*, the value is 0.384 g./cc., and in propane-methane mixtures at 61.0° C., curve *C*, 0.389 to 0.390 g./cc. The parallelism exhibited by the curves was excellent over the entire range of solute concentrations studied. In the ternary systems, however, the curves seemed to be displaced slightly towards the lower weight per cent solute concentrations. The dotted lines across the diagram show the density of liquid propane at 69.6° C. and 61.0° C. and are for comparison.

It should be noted that the densities shown by curve *A* for pure propane as

TABLE 6

SOLVENT DENSITY AT INITIAL PHASE SEPARATION

Trimyristin Weight %	Methane-Propane Density	Temp.	Propane Density	Temp.
4.05	0.361 gm./cc.	69.6° C.	----------	
8.00	0.373	"	0.370	84.3° C.
17.5	0.382	"	0.377	79.6
34.6	0.384	"	0.379	79.4
47.5	0.379	"	0.377	79.6
52.8	0.361	"	0.378	80.0
57.5	0.361	"	----------	
17.1	0.385	61.0	0.377	79.6
32.2	0.389	"	0.379	79.4
42.6	0.390	"	0.379	79.4
Stearic Acid Weight %				
3.90	0.395	88.4° C.	----------	
7.70	0.385	"	0.297	94.8° C.
19.3	0.352	"	0.321	91.6
32.4	0.332	"	0.318	92.3
41.7	0.321	"	0.290	95.2
50.6	0.298	"	----------	
17.1	0.352	71.2	0.321	91.7
29.3	0.352	"	0.322	91.5
34.2	0.307	"	0.314	92.7

--------- Indicates only one liquid phase at that solute concentration

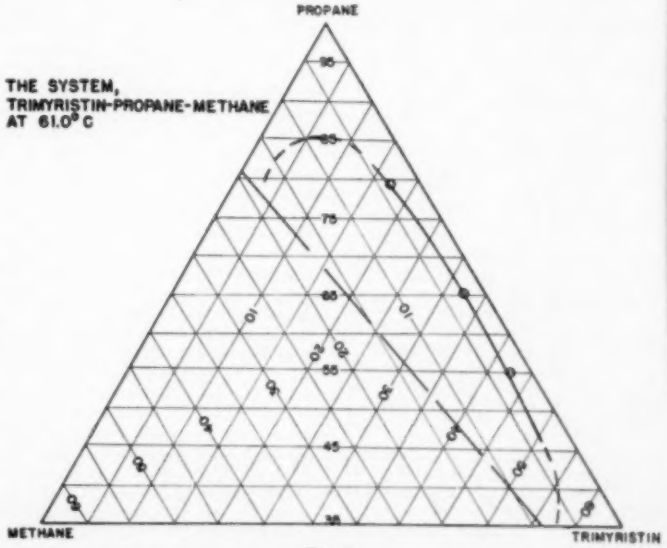

Fig. 5.

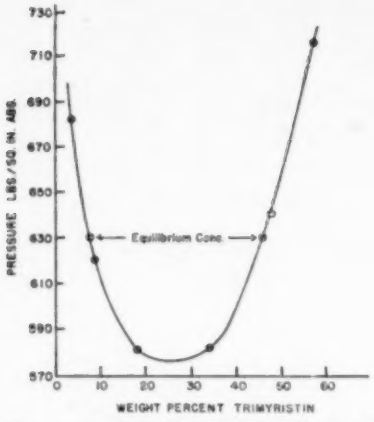

Fig. 6. The system, trimyristin-propane-methane at 69.6° C. Pressure at initial phase separation versus weight per cent trimyristin.

the solvent were obtained by using elevated temperatures. In other words, the necessary decrease in density can be obtained either by raising the temperature of the single solvent or by mixing methane with it. Although the total change in density effected by mixing with methane was only from 5 to 10 per cent, it was significant since it is equivalent to the density change produced by a 10° to 20° C. rise in temperature for propane alone.

In Figure 9, the density curve for stearic-acid-propane was obtained from the temperature-composition data of Drew and Hixson (*5*). The parallelism between the binary and ternary data at 65.4° C. was good although not as good as in the trimyristin series. For example, from curve *A*, the maximum density for phase separation with stearic acid and propane is 0.320 to 0.322 g./cc. while for the propane-methane mixtures, the corresponding value is 0.331 to 0.333 g./cc. It is obvious, however, that the densities for propane-methane-stearic acid at 71.2° C. were appreciably higher than the other two systems, the approximate maximum being 0.355 to 0.357 g./cc. In systems involving stearic acid, though, the degree of association of the acid at varying temperatures is an important factor. In general, the lower the temperature the greater the association, and therefore, the greater the effective molecular weight. If the association had been 1.8 at 90° C., perhaps at 70° C. it increased to 1.9 to 2.0. Because of the higher effective molecular weight, stearic acid should be less soluble and separate as a second liquid layer at a greater density than would be predicted. The trend of the density deviation for the ternary systems was in the correct direction.

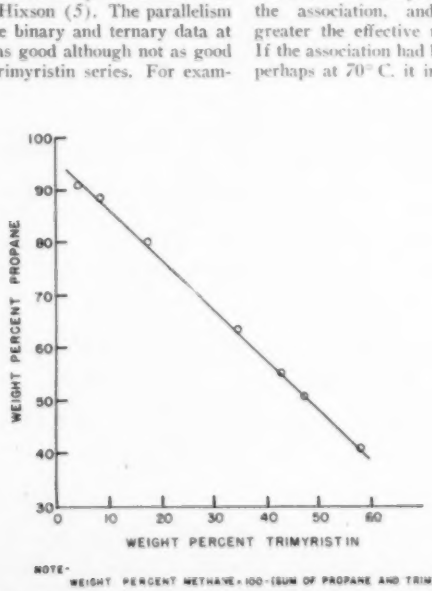

Fig. 7. The system, trimyristin-propane-methane at 69.6° C.

The solvent density, although a controlling factor, is certainly not the only variable involved in the solubility of fatty acids or triglycerides in propane-methane mixtures. The solute density also must have some effect on the solubility. Ordinarily, over small ranges in temperature the solvent density changes rapidly in comparison with the solute density. When the experimental results for the stearic acid-propane system at about 90° C. are compared to results for the propane-methane mixtures at 70° C. the changes in solute density should be considered.

In view of the fact that methane and propane are closely related members of the paraffin hydrocarbon series, the extension of the actual density values to other solvents should be done with care. This would be especially true in the case of polar solvents.

A study of the solubility of nonpolar solutes in nonpolar solvents has been made by Hildebrand and co-workers (*7*) over a ten-year period in the testing of a generalized solubility equation involving the use of both the solute and solvent molal volumes.

The equation which is based on the existence of pure Van der Waals forces between nonpolar molecules is shown as

$$\phi = V_2[(E_1/V_1)^{1/2} - (E_2/V_2)^{1/2}]^2 v_1^2 \qquad (1)$$

where

ϕ = energy of transfer of a molecule from its pure liquid to a given solution
V_2 = molal volume of solute
V_1 = molal volume of solvent
E_1 = energy of vaporization of solvent from pure liquid
E_2 = energy of vaporization of solute from pure liquid
v_1 = volume fraction of solvent in solution

When the solubilities are recorded over a range in temperature as in liquid-liquid systems E/V varies and can be replaced by a/V^2 where a represents the Van der Waals constant.

$$\phi = V_2[a_1^{1/2}/V_1 - a_2^{1/2}/V_2]^2 v_1^2 \qquad (2)$$

The term within the parenthesis often has been called D for the sake of convenience.

Since ϕ in Equation (*2*) is a measure of the energy of solution, then the solvent and solute density (proportional

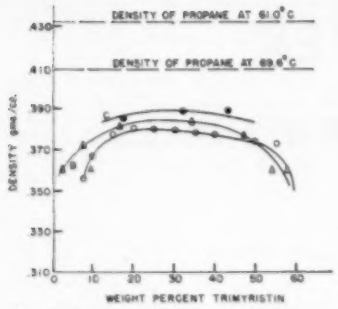

Fig. 8. Solvent density at initial phase separation for trimyristin.

A—in liquid propane.
B—in propane-methane at 69.6° C.
C—in propane-methane at 61.0° C.

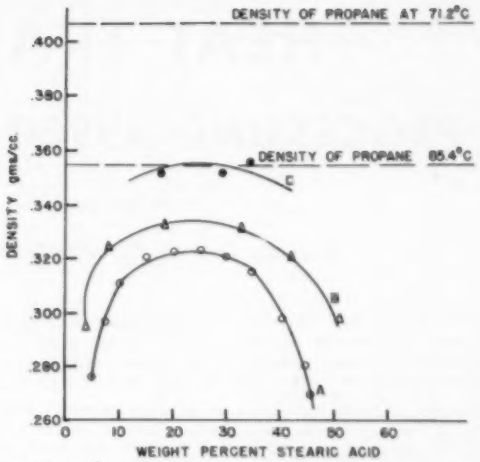

Fig. 9. Solvent density at initial phase separation for stearic acid.

A—in liquid propane.
B—in propane-methane at 85.4° C.
C—in propane-methane at 71.2° C.

to the reciprocal of the molal volume V) are the major factors affecting the solubility at a given concentration. The experimental data for the propane and propane-methane solutions seem to substantiate this contention. Furthermore, on the basis of Equation (*2*) it is easy to illustrate qualitatively why propane and propane-methane solutions exhibit a lower critical solution temperature. As the temperature increases, both the molal volume of the solute and the volume fraction solvent become greater. Over the temperature range studied the term $(a_2^{1/2}/V_2)$ representing the solute, changes only slightly, while the term $(a_1^{1/2}/V_1)$ representing the solvent, decreases rapidly. Inasmuch as the former is always greater than the latter, the net result is a substantial increase in the squared value of D. The over-all effect of all of these changes is a continuous increase in the energy necessary for solution until finally phase separation occurs. In contrast, the systems studied quantitatively by Hildebrand had a decreasing value for the term D with increasing temperature, thus diminishing the energy of transfer. Complete miscibility of two liquid phases could, therefore, be obtained by increasing the temperature. This gives rise to systems with an upper critical solution temperature.

Applications of Data. Experimental data show that phase separation for long chain fatty acids or their triglycerides can be effected by the addition of methane to propane solutions at almost any desired temperature. The same results were achieved by the addition of methane to isobutane solutions of trimyristin. Undoubtedly a similar effect could be produced by the addition of ethane to propane or isobutane although larger amounts might be necessary.

The possibility of operating a separation process at a selected temperature opens new opportunities for increasing the selectivity of the solvent mixture. Ralston and Hoerr (*10, 11, 14*) observed in solubility studies with fatty acids and a large number of nonpolar solvents that the solubility at a given temperature was primarily a function of the melting point of the acid. As the melting point of the acid is approached, the solubility increases rapidly to an infinite value. Therefore, by choosing a temperature at which one of the acids is a solid and the other a liquid, a given solvent should have increased selectivity for the fatty acid or triglyceride. This procedure can be followed by adding sufficient methane to the propane solution of the fatty acids and triglycerides to create a second liquid phase at the desired temperature.

A second advantage in the use of a solvent mixture is the added flexibility in the operation of a continuous liquid-liquid extraction system. A pressure gradient could be maintained over the system which would produce different methane concentrations. With different compositions there would be different solvent densities. Individual solutes would then tend to concentrate at specific portions of the system and could be removed as sidestreams.

Finally, the solvent mixture involves the use of propane with methane or possibly ethane. All are readily available, low-cost solvents. The chief disadvantage of the mixed solvent is the necessity of higher pressures, 700 to 800 lb./sq.in. instead of 500 to 600 lb./sq.in. An accurate economic balance would be required to determine whether or not the solvent mixture is advantageous. However, as pointed out previously, the mixture offers the attractive possibility of better separations.

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HEAT TRANSFER

PROCESSING VISCOUS MATERIALS

E. PERRY and F. E. REESE

Monsanto Chemical Company, Springfield, Massachusetts

Heat transfer in viscous materials is important in those industries processing lube oils, waxes, asphalts, high polymers and similar materials. The problem of heat transfer in the plastics industry is typical of those encountered in the general field of high viscosity products.

A theoretical approach to heat transfer in high polymers is complicated by the peculiar flow characteristics, the non-Newtonian nature, the temperature vs. viscosity relationships, and the low thermal conductivities of most polymers. This difficulty in developing a theoretical approach complicates equipment design and the development of processes.

In most cases heat transfer governs the method of processing employed. Special types of polymerization systems, e.g., suspension, mass, emulsion, and solution, are used to meet varying heat-transfer conditions. Wide varieties of polymer-processing equipment are in use to handle material in large masses or in thin films depending upon heat-transfer requirements.

The authors are unable to clarify the theoretical aspects of heat transfer for the subject class of materials, but a series of case histories is presented illustrating various methods by which satisfactory heat transfer is obtained in the processing of high polymers. Through a careful study of the chemical and physical characteristics of a material, the correct processing approach from a heat-transfer viewpoint can be selected for most viscous materials.

AN examination of the literature pertaining to heat transfer in viscous materials discloses that discussions and studies have been restricted generally to the lower viscosity ranges typified by the more fluid of the petroleum and vegetable oils. Theoretical approaches for carrying out heat-transfer calculations have been established for such materials, particularly where the fluid is being handled in pipes (*1-3, 5, 7, 9-13, 15-18*). These treatments are adequate for the majority of the heat-transfer cases which fall in the viscosity ranges below 2-5 poises.

The handling of heat-transfer problems for materials of greater than 2-5 poises viscosity unfortunately has not been equally well defined but has been characterized by references (*10*) as a "most difficult and important problem." Difficult though this heat-transfer problem may be, it still must be faced regularly for substances with viscosities of 100, 1000, or even 1,000,000 poises in those industries which process lube oils, waxes, asphalts, high polymers and similar materials. Further, the problem must be faced at present without the aid of theoretical or empirical formulas for process development or equipment design work.

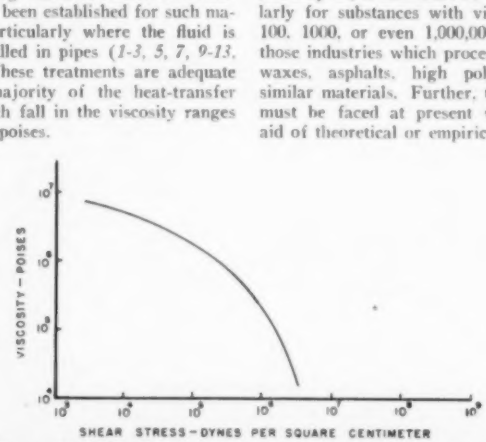

Fig. 1. Viscosity of styrene plastic as a function of shear stress.

Problems of heat transfer encountered in the high polymer field (or as it is more popularly known—the plastics field) are characteristic of those encountered in the general field of high viscosity materials. Consequently, a consideration of the heat-transfer problems and techniques in the high polymer industry is representative of the broad field of viscous materials. The polymerization and the subsequent processing of polymeric materials require adequate heat-transfer control over fluids having viscosities below that of water to those exceeding 10^6 poises—often within a single reaction step. Table 1 lists viscosities for various polymer and polymer-forming substances as an indication of the wide range of viscosities for the materials in which the high polymer industry must control thermal energy transfer. Particular emphasis has been placed in this discussion upon the viscosity range above 2-5 poises because lower viscosity materials, in most cases, are subject to analysis through available literature sources.

General Comment

A rigorous theoretical approach to heat transfer in the formation and processing of polymers is complicated by the non-Newtonian viscosity behavior and attendant peculiar flow characteristics, the temperature vs. viscosity relationships, and the low thermal conductivities of most polymers.

A detailed discussion of the rheological behavior of viscous materials (*6*) is beyond the scope of this paper. However, certain flow characteristics for the majority of high viscosity materials must be understood and borne in mind in any discussion of heat-transfer

TABLE 1.—VISCOSITIES OF TYPICAL HIGH POLYMERS AND POLYMER-FORMING MATERIALS

Material	Approximate Viscosity at 25° C.—Poises
Styrene monomer	10^{-3}
Styrene plastic	10^{14}
Phenol-formaldehyde (aqueous) mix.	10^{-2}
Phenol-formaldehyde resin	10^{10}
Vinyl chloride monomer	10^{-3}
Vinyl chloride polymer	10^{14}

mechanisms. Polymers do not exhibit a Newtonian viscosity relationship whereby the viscosity is constant regardless of the rate of shear in the material. Instead, the apparent viscosity of a polymer tends to decrease as the rate of shear increases. This effect is marked at all but low rates of shear. Figure 1 illustrates the relationship between viscosity and shear stress for styrene plastic (*14*). This pseudoplastic behavior is typical of polymers even in many cases where the polymer is present in a solution.

For highly viscous materials it is obvious that flow must be streamlined rather than turbulent. However, the type of flow exhibited in the more viscous regions is not streamlined in the conventional concept of the term. Figure 2(*a*) represents the velocity distribution exhibited by a Newtonian material in streamline flow passing through a cylindrical section. Figure 2(*b*) illustrates qualitatively the velocity distribution of a viscous polymer flowing under the same conditions. It will be noted that the flow of plastics takes place in pipes or other sections in "plug" flow wherein the central core of the material moves forward at a uniform speed and the drop-off in rate to zero at the wall is rapid over a short percentage of the distance from the wall to the center of the plug. This behavior of the material approaches that of true plastic flow where the substance has a yield point requiring a minimum force which must be exerted to induce flow of the material (*6*).

Polymers can exhibit also thixotropic characteristics wherein at high rates of constant shear there is a decrease in viscosity with time because of disentanglement and alignment of the long polymer chains.

The viscosity of a polymer is extremely dependent upon the temperature of the material. For purposes of approximation, viscosities follow the generalized viscosity-temperature curve in Figure 3 (*16*). The slope of this curve in the high viscosity regions indicates a great sensitivity of the viscosity to small temperature changes. Figure 4 presents specific data relating the viscosity of styrene plastic and of styrene plastic dissolved in styrene monomer to temperature (*14*). Wide shifts in viscosity levels with changes in temperature are typical of not only the pure polymers but also of the solutions of the polymers.

TABLE 2.—THERMAL CONDUCTIVITY OF TYPICAL HIGH POLYMERS AND OF INSULATING MATERIALS

Material	Thermal Conductivity B.t.u./(hr.)(sq.ft.)(° F./in.)
Plasticized polyvinyl butyral	0.69
Phenol-formaldehyde resin	1.01
Acrylic plastic	1.45
Polyethylene	2.32
Plasticized polyvinyl chloride	1.0
Styrene plastic	0.87
Cellulose acetate	1.75
Kaolin insulating brick	1.80
Asbestos	0.5–1.6
Fiber insulating board	0.34

Most polymers have low coefficients of thermal conductivity in the range of insulating materials such as ground cork and asbestos. Table 2 presents thermal conductivity data for several typical polymers and insulating materials. Solutions of polymers will vary in this property in relation to the type of solvent and the concentration of the polymer.

Further complicating attempts to establish valid fundamental relationships for heat-transfer calculations is the fact that the procuring of data on the above-indicated properties is a tedious and sometimes a seemingly impossible task. Influencing the values for these data are the chemical make-up of the polymer, the presence of modifiers, and the test methods used.

Polymeric materials do not necessarily possess an exact chemical composition. One of the most important fundamental characteristics governing their flow properties is molecular weight. Conventional molecular-weight determinations establish an average weight and the material is then characterized by this figure. However, all commercial polymers are mixtures of high and low molecular-weight materials, and the distribution of these values may vary widely while still maintaining the same average weight. Consequently, supposedly similar materials with identical average molecular weights may have widely varying molecular-weight distributions with resultant wide variations in flow properties.

Polymers, in common with the majority of the other highly viscous materials, are frequently combined with modifying agents during their processing. Each change in type or amount of modifier essentially results in a completely new set of physical characteristics for the polymer formulation. The number of formulations encountered in commercial practice for a given polymer is often staggering as a result of the incorporation of as many as ten modifiers in amounts ranging from 1% to 90% of the resulting product.

Finally, and of basic importance to the situation, test methods have not been established for determining the flow properties of all viscous materials, particularly in the viscosity range over 100 poises, and the validity of certain test methods which are used is open to question.

A most difficult and tedious characteristic to establish is the viscosity-temperature-rate of shear relationship. Viscosimeters used for this purpose include the Ostwald, the falling ball, the rotational, the Bingham, and the parallel plate with the latter three being particularly applicable to the higher viscosity ranges. Establishment of the required families of curves for viscosity, temperature, and rate of shear is a laborious task with any of these test methods.

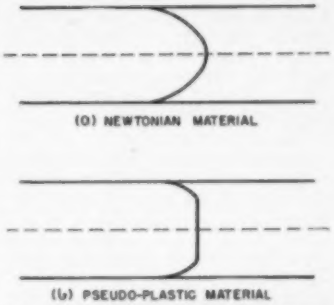

Fig. 2. Velocity distribution for streamline flow of viscous materials.

Interpretation of the experimental results obtained in viscosity determinations in the regions of high rates of shear is open to considerable theoretical question because of the previously mentioned pseudoplastic flow of polymers. Valid quantitative characterizations of viscosity for high viscosity polymers can be established only in the regions of low rates of shear. Therefore, it should be noted that viscosities indicated for specific materials in this discussion refer to viscosities extrapolated to zero rates of shear, and these viscosities are used to indicate relative viscosities for a given condition recognizing that changes in temperature and rates of shear will not produce identical shifts in viscosity levels.

Ignoring theoretical problems in the interpretation of experimental data, other operational problems make difficult the accumulation of data. During viscosity tests at significant rates of shear the material itself is apt to undergo changes which invalidate the data obtained. Mechanical shear can sever the

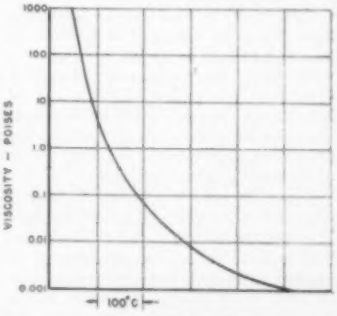

Fig. 3. Generalized viscosity curve for liquids.

bonds in some of the high molecular-weight chains with a resultant lowering of the average molecular weight and a significant and permanent decrease in the viscosity of the polymer. Thixotropic effects will also become apparent at high rates of shear.

Attempts to obtain high temperature data are often balked by the thermal instability of the material under test. Attaining of and equalization of temperatures in the test equipment may exceed the allowable residence time of the polymer at test temperatures without degradation of the polymer. Uncertain temperature control may exist in the test specimen because of heat added to the material through the energy used for producing the required rates of shear. At high viscosities even low rates of shear produce appreciable changes in stock temperature such that the establishment and maintenance of a uniform test temperature are difficult.

Despite the above-indicated problems in acquiring fundamental viscosity or flow data, progress has been made in recent years in developing versatile pieces of test equipment which can be used to establish qualitative relationships in the high viscosity ranges and even quantitative values for selected polymers under idealized conditions. It is anticipated that if the rheologists continue their present rate of progress, data will be available soon to permit precise studies of heat transfer even in the high viscosity ranges.

The authors are unable, and will not attempt, to clarify the theoretical aspects of heat transfer for the subject class of materials, but it is their intention to present a practical approach to the problem through an analysis of the fundamental factors involved in specific problems of heat transfer in high viscosity polymers. A series of case histories will be presented covering a variety of circumstances to illustrate the relation between the physical and chemical properties of a material and the solution of the heat-transfer problem for that material.

The types of heat-transfer situations which may be encountered involve the addition of heat to initiate reactions, the addition of heat to accomplish melting or fusion, the removal of heats of reaction, the removal of heat resulting from mechanical working, and the removal of heat to stop reactions or to gain rigidity in the material.

Generally, addition of heat to initiate reactions is carried out on systems having low viscosities, i.e., < 2 poises, and the problem is not too complicated at these low viscosity levels. The addition of heat to accomplish melting or fusion of materials is ordinarily associated with the most viscous of substances and presents a problem in the maintenance of an adequate rate of transfer to obtain rapid fusion.

TABLE 3.—HEATS OF POLYMERIZATION AND VAPORIZATION FOR COMMON MONOMERS

Monomer	ΔH_p-Polymerization (B.t.u./lb.)	ΔH_v-Vaporization (B.t.u./lb.)	$\frac{\Delta H_p}{\Delta H_v}$
Ethylene	1550	207	7.5
Styrene	310	150	2.1
Acrylonitrile	580	265	2.2
Vinyl acetate	440	163	2.7

The removal of heats of reaction must be accomplished from materials in all viscosity levels and represents a most perplexing step in all polymerization reactions. Table 3 lists typical heats of polymerization for several of the more common monomers to indicate the magnitude of the problem. The removal of heat resulting from mechanical working of the stock vies with heat of polymerization removal for the position of the most difficult heat-transfer problem. Mechanical shear will add appreciable quantities of thermal energy only to the more viscous fluids. Therefore, viscosities in excess of 100 poises are encountered when mechanical heat is a factor; unfortunately, the magnitude of the heat-removal problem is directly related to the viscosity encountered.

The removal of heat from materials to stop reactions (either polymerization or degradation) or to gain rigidity in materials is necessary for materials of all viscosity ranges and the difficulty of the task is a function of the substance's viscosity.

Generally speaking, the addition of heat to a viscous material does not present the problem that removal of heat presents. In the addition of heat, the material adjacent to the heating surface will be of a relatively low viscosity and the wall film can be kept to a minimum. The principal problem revolves around removal of the wall film coupled with blending it into the main body of stock. This wall-film removal supplies fresh material to the heating zone and prevents the existence of a stagnant hot layer having high heat-transfer resistances. In the higher viscosity ranges the possibility of adding heat by mechanical working provides an efficient and rapid heating scheme where applicable.

Heat transfer in viscous materials is particularly difficult when heat must be removed. The problem of heat transfer in a cooling step resolves itself into a question of how best to minimize the effect of the high film resistances on the viscous liquid side. The low thermal conductivity of the polymers means extremely high resistances for films of even moderate thickness. Also, attempts to employ high driving forces for heat transfer by using low temperature cooling media result in thick, extremely high viscosity wall films which are difficult to remove.

The general aims in cooling are to maintain a minimum film thickness through adequate removal of the wall film and to avoid the excessive addition of thermal energy from mechanical working in the higher viscosity regions.

The importance of maintaining thin films of the viscous material relates to the conventional approach to the over-all heat-transfer coefficient. If one considers a heat-transfer medium, a dividing barrier, and a material being heated or cooled all in series, then

$$U = \frac{1}{\frac{1}{h_m} + \frac{L_b}{k_b} + \frac{1}{h_p}}$$

where

U = over-all heat-transfer coefficient
h_m = film coefficient of heat transfer for the heat-transfer medium
h_p = film coefficient of heat transfer for the material being heated or cooled
L_b = thickness of the dividing barrier
k_b = thermal conductivity of the dividing barrier

The h_p is some inverse function of the film thickness and a direct function of the film's thermal conductivity. For most viscous materials, h_p is normally small compared to h_m and L_b/k_b because of the low thermal conductivity and/or the thick wall films existent in the viscous systems. Therefore, h_p is the dominant resistance to heat transfer. By reducing the film thickness, however, h_p can be increased so as to be of the same order of magnitude as the other resistances or sometimes can be increased to the point where its resistance is negligible compared to the total resistance to heat transfer.

A study of the high polymer industry immediately discloses that heat transfer almost invariably governs the processing method selected for a material. It would be easy to design equipment which has the ability to carry out the necessary mixing, distilling, or reaction steps if heat transfer could be ignored. However, heat transfer is so complex and difficult to control that all other factors are compromised to gain the necessary temperature control.

The difficulty in developing a theoretical approach to the heat-transfer problem complicates equipment design and the development of methods of processing in the high polymer industry. Equally disconcerting is the lack of universally accepted practical solutions to the heat-transfer problem. For a specific case one often finds several "preferred" methods. Unfortunately, too often it is discovered that the recommended processing step is based upon the use of a favorite type of processing equipment rather than upon a careful

study of the factors involved to single out a truly correct approach to the problem.

Examples

In the illustrations given in this section of the paper, it will be noted that the difficulty of a heat-transfer job is reflected in the size of the batch or volume which is handled. Easily controlled materials are handled in large volumes and thick sections. Into this category fall low viscosity materials, viscous media in adiabatic operation, and viscous media in which the heat load is small. With an increase of heat load or viscosity, or when limited dwell time at high temperatures is necessary, the volume of the material in process is reduced until thin films are handled in the most exacting cases. Special techniques are employed to overcome heat-transfer problems with material in the viscosity range of 10^{10} to 10^{14} poises so as to take advantage of the use of equipment with large working volumes.

I. *Large Working Volumes.* Polymerization reactions involve viscosity ranges which may be as wide as 10^{-2} to 10^{20} poises in the course of a single reaction. This change in viscosity follows from the fact that polymers, which have a molecular weight of 50,000 to 3,000,000 are produced from simple molecules of molecular weight 20 to 150. Heat-transfer problems involve both heating and cooling. Heat is supplied to initiate the reaction; during the exothermic polymerization reaction, heat must be removed; then, heat is supplied for removing undesirable volatiles and to provide sufficient mobility for handling; finally, the plastic masses are cooled for crushing and to maintain the desired shape.

Addition Polymerization

Formation of styrene plastic from styrene monomer provides an example of the problems which are encountered in polymerization reactions. Figure 4 shows how the viscosity varies widely as a function of temperature and degree of conversion. The sequence of operation involves (1) heating the monomer (10^{-2} poises) to the reaction temperature of 80 to 160° C., (2) maintaining the reacting mass (10^{-1} to 10^{12} poises) at 80 to 160° C. by removing the heat of reaction (310 B.t.u./lb.), and (3) cooling the polymer to 25° C. (10^{20} poises). Controlled partial polymerization may be effected in a kettle which is provided with a jacket and agitator, preferably with scraper blades on the heat-transfer surface to remove viscous organic wall films (*4*). Figure 5 shows how the over-all jacket heat-transfer coefficient, U, decreases with increasing viscosity for styrene polymerization in a 200-gal. kettle. At 20-40% conversion, the viscosity is so high that adequate agitation is no longer feasible and temperature control would not be possible because the semisolid mass has a thermal conductivity of 0.070 B.t.u./(hr.)(ft.)(° F.) comparable to common insulating materials. Before this point is reached the syrup can be discharged into cans of suitably small diameter (5 in. to 10 in.) such that the reaction can be kept under control by external cooling. The difficulty of providing proper heat-transfer conditions is illustrated by the fact that six hours are required to cool styrene plastic from 160° C. to 40° C. when the heat transfer distance is 2 in. and the cooling fluid has a temperature of 15° C.

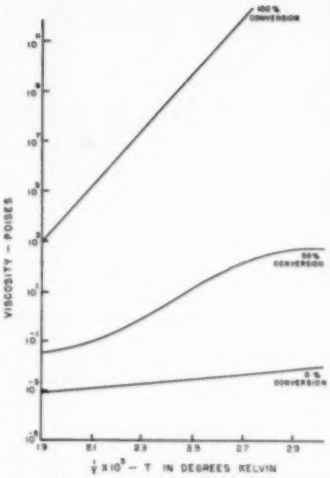

Fig. 4. Viscosity of styrene plastic solutions in styrene monomer as a function of temperature and conversion.

Condensation Polymerization

Condensation of phenol with aqueous formaldehyde to produce lump (i.e., 100% solids) resins involves the viscosity ranges of 10^{-2} to 10^{6} poises. Reaction is initiated at 50° C. and the exothermic chemical reaction is controlled between 70° C. to 95° C. After the reaction, excess water is removed by vaporization under reduced pressures as the resin temperature changes from 50 to 90° C. The resulting lump resin is cooled from 90° C. to 25° C., and may be ground for further processing convenience.

Chemical condensation and dehydration can be carried out in 500 to 3000-gal. kettles, provided with jackets for temperature control. Anchor-type agitators, equipped with scrapers, are mandatory because the resins are thermosetting (i.e., heat-hardening); excessive wall temperatures will cause the formation of insoluble, infusible films which lead to unsatisfactory conditions of heat transfer. The viscosity increases throughout the cycle and is at a maximum at the end of the dehydration. Figure 6 illustrates how the over-all jacket heat-transfer coefficient varies with time during the dehydration cycle. The rapid decrease in the coefficient after two hours coincides with the disappearance of the aqueous phase.

Kettle-cooling of the lump resin is unsatisfactory because of the time involved with the low jacket heat-transfer coefficients and because the cold mass has such a high viscosity (ca. 10^6 poises) that removal from the kettle would be difficult. Figure 7 shows how the viscosity of the dehydrated resin varies as a function of temperature. For rapid cooling, the mass can be dropped onto a water cooled floor where the resin spreads in pancake form with thicknesses of 1 in. to 4 in. Cooling is effected by contact with the floor and by recirculating air over the upper surface. Figure 8 illustrates the time-temperature cycle for a resin pancake of 4-in. thickness on a cooling floor. As an alternative, when the resin is to be shipped without further processing, it is often advantageous to discharge the dehydrated mass directly into shipping containers which are small enough for cooling within the allowable time-temperature limits. Figure 9 shows typical cooling curves and illustrates the advantage of water vs. air cooling the container. Apparent over-all heat-transfer coefficients for the two cases in Figure 9 are: Case *A*—air cooled, $U = .33$ B.t.u./(hr.)(sq.ft.)(° F.). Case *B*—water cooled, $U = .70$ B.t.u./(hr.)(° F.)(sq.ft.).

When phenolic varnishes are produced, the sequence of operations is the same as for lump resins through the dehydration stage. Instead of dumping for cooling, cold solvent is added directly to the mass in the kettle before the viscosity of the resin exceeds 10^6 poises and the viscosity of the mass is reduced by the solvent to the 1- to 10-poise range. The batch temperature is lowered by contact with the cold solvent and with the heat-transfer surface. With the working material in the low viscosity range, over-all jacket coefficients of 40-80 B.t.u./(hr.)(sq.ft.)(° F.) can be realized.

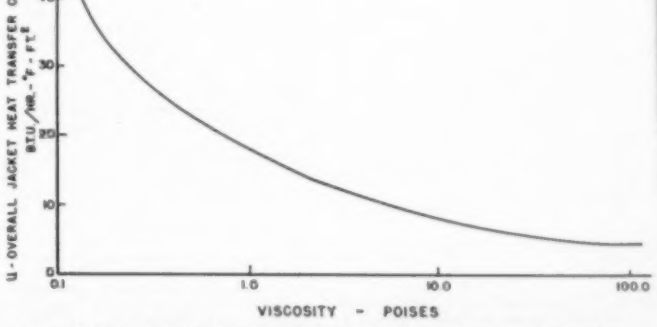

Fig. 5. Over-all jacket heat-transfer coefficient as a function of viscosity. Mass polymerization of styrene in a kettle with agitation.

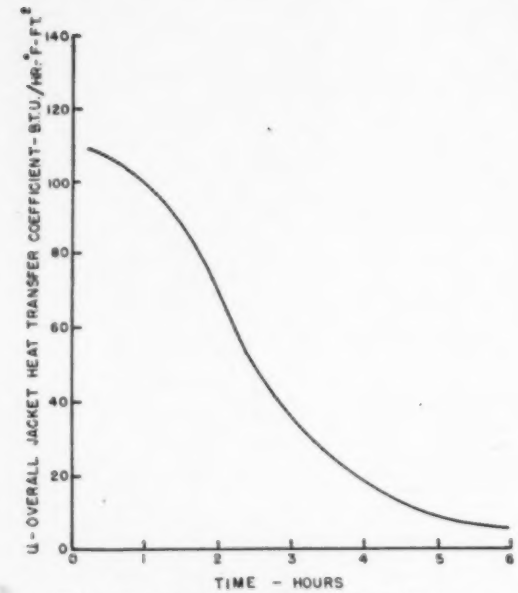

Fig. 6. Over-all jacket heat-transfer coefficient as a function of time.

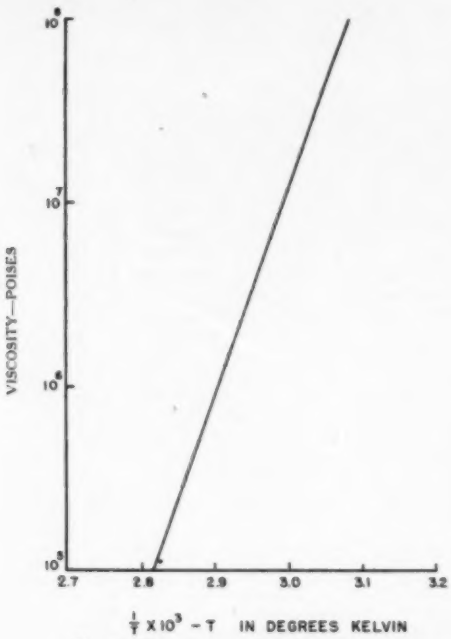

Fig. 7. Viscosity as a function of temperature —phenolic lump resin.

Blending and Colloiding

Many dissolving, heating, and blending operations are carried out with resins in the viscosity range of 10^4 to 10^8 poises. Heat input may be through the jacket or by means of dissipation of mechanical energy on the material. For materials with viscosities above 10^8 poises the dissipation of mechanical energy is most significant because heat transfer through the jacket walls is small. The greater the viscosity of the material, the more severe the problem of temperature control.

The mixing of a cellulose-acetate-type material in a 150-gal., jacketed, Baker-Perkins mixer can be used to illustrate the problems involved in mixing operations. Figure 10 presents a stock temperature vs. time curve and shows how the stock temperature gradually increases to 61° C., despite a jacket temperature of 26° C. This material has a viscosity of 10^4 poises at 50° C. By adding solvent an equilibrium level at 55° C. is established. This decrease in temperature is a function of the lower stock viscosity (10^6 poises at 50° C.) which decreases the addition of heat from mechanical working and simultaneously increases the heat-transfer coefficient through the jacket.

A parallel case in the same equipment is that of a vinyl solution in which material of 10^4 poises at 50° C. attains an equilibrium temperature of 93° C. with a 24° C. jacket temperature. By decreasing the viscosity to 10^6 poises at 50° C. through solvent addition, the equilibrium temperature falls to 43° C.

It is possible to decrease the equilibrium temperature without viscosity reduction by decreasing the paddle speeds, i.e., by decreasing the rate of mechanical energy input. Figure 10 indicates how the equilibrium temperature is reduced from 55° C. to 50° C. by changing the paddle speeds from 30 and 20 rev./min. to 25 and 15 rev./min.

Intensive mixing for the fusion and blending of materials in the range of 10^6 to 10^7 poises and 120° to 180° C. is effected often in a Banbury mixer. A widely used unit for polymer processing is the 3*A* size which handles 125-150 lb. of material. As with the Baker-Perkins mixer the most serious problems are concerned with mechanical heat input which is even greater for a Banbury because of the higher viscosity of stock being processed. In the range of working viscosities, jacket temperatures and heat transfer through the walls have no effect on the operation once the material is sufficiently fluxed to permit the intensive mixing action of the paddles to become effective. Temperature control is a function of mixing intensity alone since the operation is essentially adiabatic.

Cycles are short followed by rapid removal of the stock to avoid degradation once the maximum allowable temperature is reached. Figure 11 illustrates a typical stock temperature curve for a plasticized vinyl polymer of viscosity 10^6 to 10^8 poises at 160° C. in a number 3*A* Banbury operating at 70 rev./min. with a 140-lb. charge. The rate of temperature rise is constant

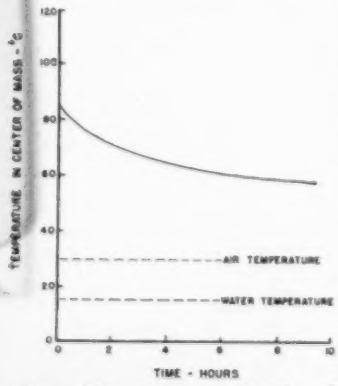

Fig. 8. Temperature as a function of time. Cooling phenolic lump resin on a cooling floor.
Resin thickness of 4 in.

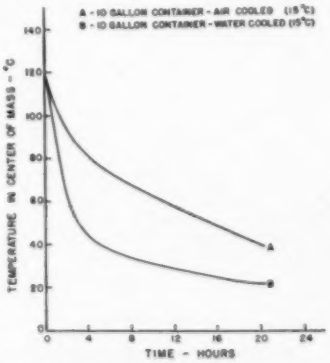

Fig. 9. Temperature as a function of time-cooling phenolic lump resin.
A—10 gal. container—air cooled (15° C.).
B—10 gal. container—water cooled (15° C.).

TABLE 4.—EXTRUSION OF STYRENE PLASTIC IN A 6-IN. DIAMETER BARREL EXTRUDER (RATE OF 500 lb./hr.)

Jacket Temperature (° C.)	Stock Temperature (° C.)
249	277
231	274
190	277
177	271
166	271

throughout the cycle and is apparently independent of the 110° C. jacket temperature. Experiments show that the principal effect of the jacket and cored paddle temperatures is to change slightly the time at which the stock temperature rise begins after charging. Once the rise begins, the jacket temperature is ineffective in controlling the rate of rise in the allowable working temperature range; in practice, after the unit is heated by operation, the heating jackets often are not utilized at all. Some control of stock temperature can be effected by controlling paddle speed. By reducing the paddle speed to 35 rev./min., the initial dwell time before the temperature begins to rise would be doubled and the rate of heating would be reduced to a value of 60% of the rate for the higher speed.

Continued temperature rise of stock in Banburys is checked by dumping the plastic to mill rolls on which the temperature can be maintained or lowered slightly for further processing (see below).

Extrusion

Mixing and fusion of material in the range of 10^5 to 10^7 poises is also effected in screw extruders. For commercial size extruders (2½-in. minimum diameter) the heat-transfer conditions are similar to those in a Banbury unit and the control of stock temperature is a function of mechanical heat input. Jacket temperatures are ineffective in the control of stock temperature as is illustrated by Table 4 for the extrusion of styrene plastic in a 6-in. diameter barrel plastics screw extruder at a rate of 500 lb./hr. Reduction in cylinder temperature of 83° C. has a negligible

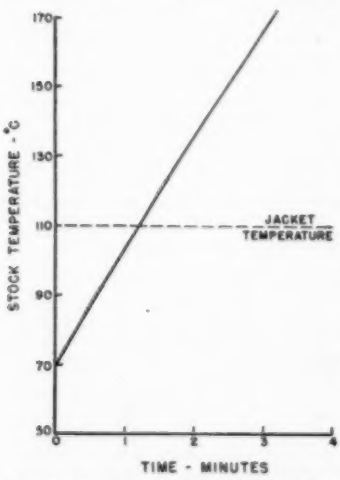

Fig. 11. Stock temperature as a function of time.
Plasticized vinyl polymer in No. 3A Banbury
70 rev./min.—140-lb. charge.

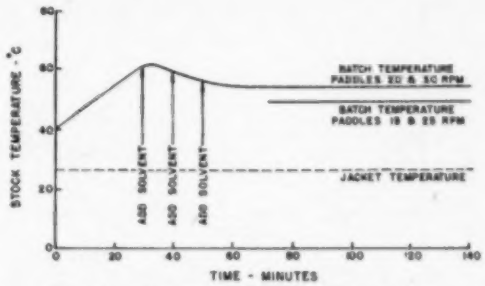

Fig. 10. Stock temperature as a function of time.
Mixing cellulose acetate formulation in a 150-gal. jacketed Baker Perkins mixer.

(6° C.) effect on stock temperature. A calculated heat balance around this machine in normal operation shows that heat input through the jacket is less than the theoretical radiation loss and that part of the mechanical heat input to the stock supplies the balance of the radiation loss. This calculation has been confirmed by insulating the machine more efficiently and turning off the heating system, whereby an increase in stock temperature was observed.

Control of stock temperature is a function of the control of mechanical heat input. Mechanical energy input can be controlled by varying the screw speed or by redesigning the screw. Table 5 illustrates the relationship between stock temperature and screw speed for a vinyl compound of viscosity 10^4 poises at 320° C. being processed in a 2½-in. diameter barrel plastics extruder. Temperature control by screw redesign is of limited value because the primary object of the operation is fusion rather than conveying of material.

For experimental laboratory work, using extruders of small bore such that the ratio of jacket area to working volume is large and throughput is small, control of stock temperature can be accomplished via jacket control.

The material leaves the extruder through a die in thin sections in ribbon or rod shape whereby cooling can be accomplished rapidly in a liquid or gaseous atmosphere.

Melting and Mixing

Production of vinyl resin-wax hot melts for paper coatings and adhesives illustrates simple melting and blending in the range of 10^3 to 10^5 poises in jacketed kettles. No chemical reaction is involved and mechanical energy input is small. Heavy-duty anchor-type agitators are required to handle the mass, especially at the low initial temperatures. Figure 12, showing the over-all heat-transfer coefficient as a function of viscosity, illustrates the typical worsening effect of a viscous organic film on heat transfer. The hot liquid is discharged from the kettle directly into shipping containers where slow cooling is permissible because the product is essentially non-heat sensitive in the working range. For example, 150 lb. of hot melt at 130° C. contained in a cylinder 18 in. in diameter by 20 in. high requires 16 hr. to cool to 40° C. when stored in a room at 25° C. The apparent over-all heat-transfer coefficient under these conditions is 0.56 B.t.u./(hr.)(° F.)(sq.ft.).

Forming

Shaping and forming of polymeric masses are important operations in the plastics industry. For this purpose, the material is heated just high enough to allow flow so that it will weld to itself under the operating pressures. Once in the desired shape, the mass is cooled so that it will not deform on further handling. The heat load may not be great but the heat-transfer problems in large masses at 10^6 to 10^8 poises working viscosity are difficult because of the low thermal conductivity of polymers.

The cake-baking method for the manufacture of cellulose nitrate sheet stock illustrates a typical forming operation. Stock, which has been compounded on mill rolls is slabbed into rectangular sheets 23 in. × 56 in. × 1 in. and placed to a depth of 6 in. in a thick steel frame together with top and bottom plates which fit inside the frame. The assembly is put between the platens of a heated hydraulic press and subjected to a pressure of 200 lb./sq.in. with 90° C. water circulating in the platens. Fusion will occur when the stock temperature reaches 55° C. The polymer must be cooled to 38° C. before the pressure is released or further deformation will occur. The cold cake is sheeted by a knife to give sheets of the proper thickness.

Figure 13 shows the heat history of a cake during a cycle consisting of 90° C. water for 6 hr. followed by 5° C. water for 5 hr. in the platens. It will be noted that

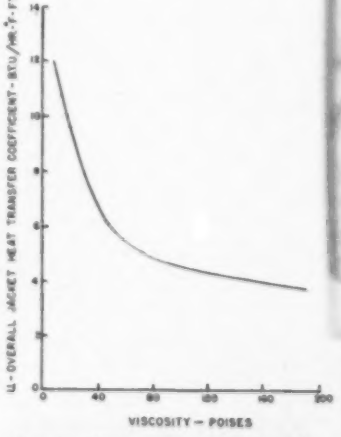

Fig. 12. Over-all jacket heat-transfer coefficient as a function of time.
Cooling of vinyl base wax hot melt in a jacketed kett'e with agitation.

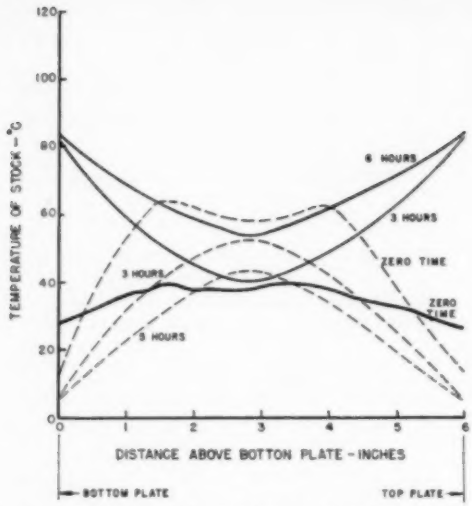

Fig. 13. Stock temperatures as a function of location and time.
Production of cellulose nitrate base cake.
23 in. x 50 in. x 6 in. cake between cored platens.
——— heating
- - - cooling

the starting temperature curve is uneven as a result of heat loss from the warm rolled stock to the cold cake-binder. The heating cycle to bring the stock to 55° C. could be shortened considerably by using higher temperatures in the heated platens were it not for the existence of a maximum stock temperature above which the nitrate plastic decomposes violently. In this particular operation, the variables are sufficiently few and well defined so that the problem can be treated by mathematical analysis (*13*). Calculated temperatures for the inner regions of the cake have been found to agree within 1-2° C. with measured temperatures.

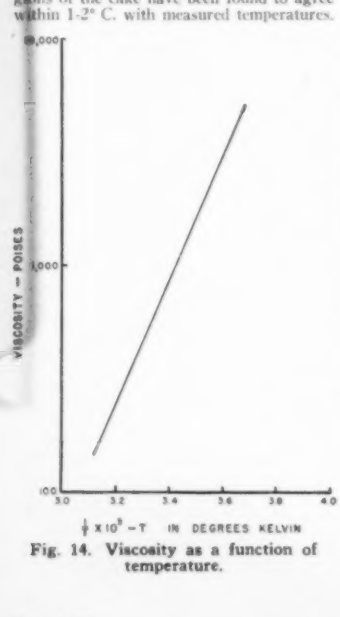

Fig. 14. Viscosity as a function of temperature.

Mill Rolling

II. *Small Sections.* Fusion and the compounding of plastics in the viscosity range of 10^2 to 10^6 poises is carried out often on mill rolls. The problem involves fusion by heating but without exposing the stock to conditions of degradation. Temperature control is accomplished by handling sections of ⅛ in. to ½ in. thickness. The milling technique was borrowed from the rubber industry and is being displaced gradually by such units as extruders and Banburys in which the material is exposed to a higher temperature for a shorter period of time.

For the initial part of the operation heat is supplied to the material by the hot rolls; once fusion begins the rolls are used for cooling because the mechanical heat input predominates. The mechanical work is done on material held in the nip between the rolls and this work is a function of the size of the nip, the speed differential between the rolls, and the absolute speed of the rolls. For example, styrene plastic processed on rolls with a 1.1:1 speed ratio and a front roll temperature of 150° C. and a back roll temperature of 95° C. reaches an equilibrium temperature of 235° C. A lower viscosity vinyl compound will reach an equilibrium temperature of 165° C. with both roll temperatures at 145° C. and the same 1.1:1 speed ratio. At a speed ratio of 1.5:1 and all other conditions unchanged, the equilibrium temperature for the vinyl stock would be 175° C. A change in the temperature of the rolls can be used to effect a change in the final level of the stock temperature, but an equilibrium stock temperature level, which is higher than the roll temperature, exists for any given set of conditions.

Molding

Molding of thermoplastic (i.e., heat-softening) resins is carried out with the material in the 10^6 to 10^9 poises viscosity range. Once formed, the plastic must be cooled so as to retain its shape. Rapid heating followed by rapid cooling is important for production capacity as well as to avoid thermal degradation at the high temperatures. Polymeric materials, with their low thermal conductivities, must be handled in thin sections in which the ratio of heat-transfer area to material volume is large. For example, the injection molding of styrene plastic is carried out by first heating the cold pellets in an annular space between an outer wall and an inner torpedo. The maximum thickness of annulus is ½ in. From the cylinder the stock flows through a mixing nozzle to equalize any temperature differences which might exist, and is injected into a die. The die is maintained at the minimum possible temperature which still gives an article without case-hardening and resulting internal-shrinkage voids. The actual die temperature is a function of initial stock temperature and the thickness of the mold. Cooling is continued until the temperature of the plastic is below the 88° C. heat distortion point. Table 6 lists typical temperatures and operating conditions for the proper cooling of molded styrene plastic as a function of section thickness.

The cooling of phenolic lump resins on cold floors described previously is another example of handling materials of viscosity 10^2 to 10^{10} poises in thin sections.

III. *Thin Films.* When the material is so handled as to be present in layers and thin films, excellent temperature control is obtained. The advantage of thin films for temperature control was shown earlier by considering a heat-transfer medium, a physical barrier, and the material being treated in series (*see* General Comment).

Votator

The rapid cooling of a heat-sensitive phenolic varnish to prevent overreaction illustrates the handling of material in the viscosity range of 10^3 to 10^6 poises as a thin film to improve heat transfer. For example, a Votator may be used (*8*). The Votator is a machine which handles the working material in a ¼-in. annular space with the rotating central core equipped with scrapers to remove wall films and expose the heat-transfer surface continuously. Because of the increase in viscosity of the varnish as the temperature is reduced (*see* Fig. 14) a thick organic wall film forms and hinders heat transfer in conventional kettle equipment. Figure 15 contrasts the over-all heat-transfer coefficients obtained as a function of resin viscosity for Votator and kettle operations. Cooling from reaction temperature to storage temperature in the Votator requires one twentieth the time required in the kettle.

Drum Cooling

The cooling of thermoplastics of low thermal conductivities in the viscosity range 10^3 to 10^{10} poises can be effected rapidly in the form of thin films. For example, cooling drums may be used as in the formation of flexible vinyl sheet of 0.015-in. thickness. Stock at a temperature of 168° C. being fed to a cooling drum with a surface temperature of 77° C. leaves the drum at 88° C. after a contact time of 7.34 sec. The apparent film heat-transfer coefficient in this case is 22 B.t.u./(hr.)(sq. ft.)(° F.). If the vinyl film has an initial temperature of 77° C. and the drum surface temperature is 13° C., the ribbon discharge temperature is 16° C. after a contact time of 16.1 sec. Under these conditions, the apparent film heat-transfer coefficient is 11 B.t.u./(hr.)(sq.ft.)(° F.); this lower value

TABLE 5.—EXTRUSION OF VINYL COMPOUND IN A 2½ IN. DIAMETER BARREL EXTRUDER

Worm Speed (rev./min.)	Stock Temperature (° C.)
70	221
60	210
50	199
30	182

reflects the poorer contact of the colder, stiffer ribbon with the drum surface.

Drum Drying

Heating of viscous solutions of plastic materials to remove volatile diluents and to make products in the viscosity range of 10^{20} to 10^{22} poises presents a problem of heat transfer and resin stability which can be solved only by utilizing films of 0.0001 in. to .003 in. in thickness. For example, in the drum-drying of a particular plastic material the contact time of the solution with the drum of 1½-in.-shell thickness varied from 5 to 15 sec., the heating medium was at 171° C., and the plastic was discharged from the drum at 104° C. Under these conditions the apparent over-all heat-transfer coefficient was 140 B.t.u./(hr.)(sq.ft.)(° F.). The major resistance to heat transfer existed in the drum shell, i.e., the barrier which, for most heat-transfer problems, can be considered negligible. Table 7 illustrates how the distribution of the resistance to heat transfer varied as a function of the film thickness for this operation and illustrates the importance of maintaining thin films.

IV. *Special Techniques.* Special techniques for meeting the heat-transfer problems involved in processing viscous materials include (a) dilution with solvent, (b) subdivision whereby heat-transfer distances are reduced even though the material is still handled in large capacity equipment, and (c) vaporization.

Dilution

Dilution with solvent has a threefold beneficial effect: (1) reduces viscosity to improve jacket coefficients, (2) mixes cold fluid with the hot mass by direct contact, and (3) adds heat capacity to the system. Rapid cooling by dilution can be effected by cold monomer addition to polymerization reactions which tend to get out of control, providing the monomer and polymer are completely miscible. The polymerization of vinyl acetate represents such a case. In the polymerization of ethylene, a dilution method is required because (1) of the gaseous phase polymerization in which film coefficients are small (2) of the high heat of polymerization per unit weight of polymer formed (1800 B.t.u./lb.) and (3) of the resistance to heat transfer of the thick metal walls required at the reaction condition of 15,000-30,000 lb./sq.in. and 150 to 250° C. The problem of temperature control is overcome by obtaining a low conversion of ethylene per pass and utilizing the heat capacity of the total throughput to limit the temperature rise. Even under such conditions temperature control is difficult and the heat rise in the reactor is of the order of 100-200° C.

TABLE 6.—INJECTION MOLDING OF STYRENE PLASTIC

Section Thickness (in.)	Stock Temperature (° C.)	Die Temperature (° C.)	Set[1] Time (sec.)
1/16	288	50	15
1/8	260	60	30
1/4	232	70	45
1/2	182	80	90

[1] Time for surface temperature to fall below 88° C. and interior temperature to be such that voids and distortion are avoided.

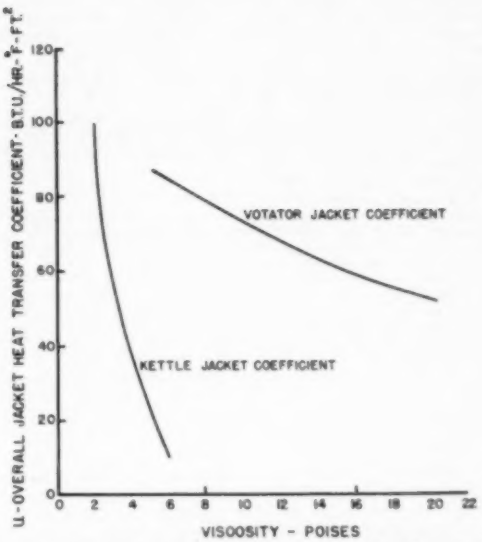

Fig. 15. Over-all jacket heat-transfer coefficient as a function of viscosity. For phenolic varnish A.

Direct dilution with cold water may be used to check phenolic resin reactions in the 10^5 to 10^8 poises range if the water can be decanted at a later stage of the reaction. If the viscosity is too great before the addition of water, dilution is difficult because of agitation difficulties. When only slight dilution is permissible, the high heat of fusion of ice (144 B.t.u./lb.) can be utilized by direct addition of ice to the solutions in the viscosity range of 1-100 poises where satisfactory agitation is still possible. If no water can be tolerated, solid carbon dioxide can be added. Since the carbon dioxide evolved serves to agitate the mass, "dry ice" cooling can be utilized in those viscosity ranges where efficient mechanical agitation fails (10^6 to 10^8 poises). In general, cooling through barrier walls is to be preferred whenever it can be accomplished from the heat-transfer point of view because (1) contamination is avoided and (2) the operational cost is less once the capital expenditure has been provided.

A special and rarely used case of dilution is illustrated by polymerization in the presence of a material which is a solvent for both the monomer and polymer such that the polymer solution is maintained at a low viscosity. The disadvantages of solution polymerization are (1) high cost due to solvent loss and recovery and (2) viscous conditions ($>10^4$ poises) which are encountered at some stages of the reaction unless the solids are so low as to be out of the commercially feasible range of utilization (*see* Fig. 4).

Subdivision

Solvent, nonsolvent polymerization technique, a borderline case between the dilution and subdivision approaches, was developed to overcome the disadvantage of the high viscosity of dilute polymer solutions. In this technique the monomers are soluble in the solvent but the polymer is not. Therefore, as the polymer is formed, it precipitates from the solution as a finely divided solid and forms a slurry. Satisfactory conditions of heat transfer are obtained which can be treated by conventional methods for low viscosity (< 1 poise) liquids, but the cost of solvent-handling places a severe restriction on the extensive use of solvent, nonsolvent polymerization processes. Polymerization of vinyl chloride in mass (i.e., with no diluents) is illustrative of the solvent, nonsolvent technique.

Suspension polymerization, carried out in an aqueous medium, offers the same advantages of heat transfer as the solvent, nonsolvent technique, but without the disadvantage of high solvent costs. In suspension polymerization, neither the monomer nor polymer is soluble in the medium. At the beginning of the reaction liquid monomer droplets of less than 10^{-2} poises viscosity are suspended in water by means of suitable agitation. Polymer forms in the course of the reaction in small spheres 1/32 in. to 1/64 in. in diameter so that a slurry is obtained. The heat of polymerization is removed easily from the droplets even when the solids content of the droplets approaches 100% and the viscosity reaches 10^{10} poises because the maximum heat-transfer distance is on the order of 1/64 in. to 1/128 in. The temperature of the aqueous suspension medium is controlled by conventional methods for low viscosity (< 1 poise) fluids. The suspension polymerization of styrene is illustrative of plastic materials which can be polymerized commercially by the suspension technique. The over-all jacket coefficient of heat transfer in large commercial metal reactors has been mea-

TABLE 7.—DISTRIBUTION OF RESISTANCE TO HEAT TRANSFER IN A DRUM-DRYING OPERATION

Film Thickness of Product (in.)	Per Cent Resistance in		
	Heat Transfer Medium	Barrier	Plastic Film
0.000400	14	69	17
0.000430	13	67	20
0.000476	12	63	25
0.000530	11	58	31
0.000550	10	55	35
0.001000	7	25	58

sured at 90 to 110 B.t.u./(hr.)(sq.ft.)(° F.) and remains constant even as the viscosity of the droplets changes from 10^{-2} poises to 10^{12} poises. This high value of the coefficient should be contrasted with those in Figure 5 which show the jacket coefficient as a function of viscosity for the polymerization of styrene without diluents.

For heat-transfer purposes, emulsion polymerization can be considered as an extreme case of suspension polymerization in which the heat-transfer distances are reduced to approximately 0.1μ. The over-all mass is maintained at a low viscosity ($< 10^{-2}$ poises) throughout the course of the reaction even though the particles may reach a viscosity of 10^{12} poises toward the end of the reaction cycle. For example, vinyl chloride can be polymerized in aqueous emulsion form as can styrene, and styrene-butadiene. Typical over-all heat-transfer coefficients have been measured for these reactions:

U glass-lined kettle jacket

40-60 B.t.u./(hr.)(sq.ft.)(° F.)

U metal-kettle jacket

90-110 B.t.u./(hr.)(° F.)(sq.ft.)

Vaporization

Heat removal by vaporization is advantageous whenever it can be utilized. The volatile vapor has a low molecular weight and low viscosity and, consequently, large values of heat-transfer coefficients characteristic of condensing vapors can be realized (U = 75-150 B.t.u./(hr.)(sq.ft.)(° F.). Correspondingly, required condenser areas are 1/10 to 1/15 of required jacket areas, and it is a simple matter to supply the area in the form of a condenser. Vaporization can be used in suspension and emulsion polymerizations to effect heat removal from the low viscosity medium at a rapid rate of reaction. Unfortunately, for viscous masses where the supplementary heat-removal facilities are required most urgently, vaporization is limited by foaming tendencies. In the viscosity range below 1 poise, removal of heat by vaporization is feasible in commercial size (> 200-gal. capacity) equipment. For special cases of a steady and unchanging heat load, where sudden fluctuations in pressure can be avoided, heat removal via vaporization is practical in the 10^1 to 10^2 poises viscosity range in 300-gal. capacity kettles. For example, the temperature of the initial condensation reaction between phenol and aqueous formaldehyde can be controlled by maintaining a suitable pressure on the system in conjunction with a reflux condenser. Where control by reflux is possible close temperature control can be effected. Table 3, illustrating the fact that 2 lb. to 5 lb. of monomer must be vaporized per pound of monomer reacting for common plastic materials, emphasizes the difficulty of the foaming problem.

Summary

The general approach for controlling heat transfer in viscous materials is as follows:

1. As a material becomes more viscous and/or the heat-transfer requirements become more exacting, the material should be handled in decreasing volume.
2. As the viscosity of a material rises, increased attention should be paid to the effects of mechanical work on the material.

Application of these rules will vary depending on whether heat addition or heat removal is required.

If heat addition is required, a large area of transfer surface per volume of material is desirable. Little or no assistance in spreading of the heat through the mass can be expected from convection currents and relatively little from conduction. Consequently, adequate removal and blending in of the heated wall films must be provided. Wall films will have high resistance to heat transfer and should be kept to minimum thickness by mechanical means. Long-dwell times for the wall film or overheating of the wall film must be avoided for heat-sensitive materials. Whenever practical, the addition of heat by mechanical working is a rapid and efficient means and should be employed for materials of more than 10^8 poises viscosity.

If the removal of heat from viscous substances is required, the same requirement exists for the area-volume relationship, for the effective maintenance of thin wall films, and for the adequate blending of the removed wall material with the main mass. As the viscosity of the material increases the amount of mechanical energy imparted to the stock for this film removal and blending must be decreased to prevent addition of heat from this source. Consequently, when wall-film-thickness control is no longer feasible by mechanical means, the material must be handled in increasingly smaller sections as the heat-removal requirements become more stringent.

In certain cases heat removal may be aided by the use of an intermediate medium to which the viscous material readily can transmit its heat, and this medium, in turn, readily give up its heat. This latter situation is exemplified by the use of diluents which can be evaporated for heat removal or by the use of low viscosity media in which the higher viscosity materials can be dispersed in small volume particles.

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Discussion

W. R. Marshall, Jr.: I would like to know what the conditions were for the estimation of the resistances. Normally, in any drying process, you have a period when you have surface moisture evaporating and a falling rate. Furthermore, in drum-drying you can presumably come up with a range of final moisture contents. Now that means the heat-transfer rates and resistances would have to be determined either for some average temperature of the material, or for a given period of drying. Can you tell us what the conditions were for these resistances and how you determined surface temperatures if you did so?

E. Perry: I can give you some information about that. As Professor Marshall points out, our over-all coefficients were, of course, average coefficients. We were going from an aqueous mixture to essentially a dry solid. We had operating data, and we calculated our coefficients and compared the results as calculated and as measured. They agreed, and we concluded that the calculations were sound.

W. R. Marshall: In other words they covered the entire range of drying? You had essentially an average material temperature which might be approximately constant, and then would be rising toward the end of the cycle.

E. Perry: As far as the material temperature is concerned, it was essentially constant. For this particular aqueous mixture, boiling occurred at 98-100° C., and the dry material from the drum was about 5 or 10° lower in temperature.

W. R. Marshall: What was the moisture content of the dry material?

Mr. Perry: Well below ½%.

(*Presented at Forty-third Annual Meeting, Columbus, Ohio.*)

CONTINUOUS FLOW STIRRED TANK REACTOR SYSTEMS

Agitation Requirements

R. W. MacDONALD† and EDGAR L. PIRET

University of Minnesota, Minneapolis, Minnesota

Development of the theory and practice of continuous blending and of continuous flow stirred tank reaction systems is of increasing industrial importance. To date, however, little or no experimental or theoretical work has been available on the problem of providing satisfactory intensities of agitation for such operations.

The time required for complete distribution throughout a continuous flow stirred reactor of a portion of dye injected into the feed stream has been measured as a function of agitator speed and feed stream rate. A critical agitator speed for mixing time has been demonstrated. A tentative method of correlation is suggested which is based upon the energy input of the agitator plus that of the feed stream.

A first-order continuous flow reaction was studied in a single and also in a series of vessels, at various flow rates and agitator speeds. As the agitation intensities were reduced, the concentration of the effluent was found to fluctuate more widely from the theoretical and always in an irregular manner. It is proposed that a satisfactory intensity of agitation for a stirred tank reactor exists when the effluent concentration approaches the theoretical concentration and only fluctuates from it within the limits imposed by the needs of subsequent operations and requirements.

A CONTINUOUS flow stirred tank reaction system is one in which the reaction volume is divided into separate compartments, in each of which homogeneity is maintained by stirring. The mixed reactants are fed into the first compartment and the effluent, removed at the same rate, is in a like manner fed into the other compartments of a series (Fig. 1). Potential economics in labor costs and advantageous conditions for ready control of a production process make continuous flow reactor systems of particular interest to present-day industry. Design equations for such reactor systems have been in this laboratory extended to a wide range of reaction types and to both steady state and transient conditions of operation (*9, 25*).

In developing the theory of continuous flow stirred tank reactors the assumption is usually made that the concentration of reactants and products in each subdivision of a compartment is equal to the concentration in every other subdivision and, of course, equal to the effluent concentration from that compartment. That is, the feed solution is assumed to be dispersed "instantaneously" throughout the vessel volume. The purpose of this investigation was to study the factors involved in attaining this homogeneity. Effects of insufficient agitation on the composition of the product stream from a single and from a series of continuous stirred tank reactors were also investigated.

While Asquith (*1*) has made a point of the importance of agitation studies in continuous flow reaction systems, little information on the subject is available in the literature. The concepts developed in this work are therefore to a large extent original.

Mixing times in a reaction vessel were measured in this investigation by introducing a small volume of dye solution into the feed stream of a continuous flow vessel. The independent and combined effects of variations in agitator speed and of variations in feed rate on the time required for uniform distribution of the color throughout the vessel were determined.

The continuous hydrolysis of acetic anhydride, a first-order reaction, was carried out in a single and also in a series of continuous reaction vessels. Effects of agitator speed and volumetric feed rate on the degree of completion of this reaction were investigated and an attempt made to relate these results to the previously measured mixing times.

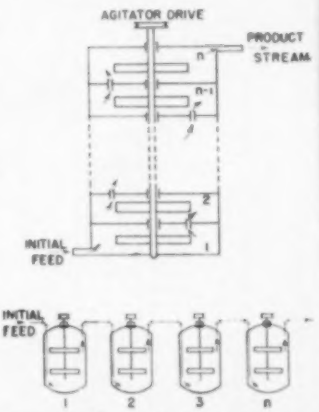

Fig. 1. Continuous feed stirred tank reactors operated in series.

Complete data for Table 1 and Table 3 which accompany this paper are on file (Document 3298) with the American Documentation Institute, 1719 N Street, Northwest, Washington, D. C. Obtainable by remitting $1.00 for microfilm and $1.00 for photocopies.

† Present address: Esso Standard Oil Co., Baton Rouge, La.

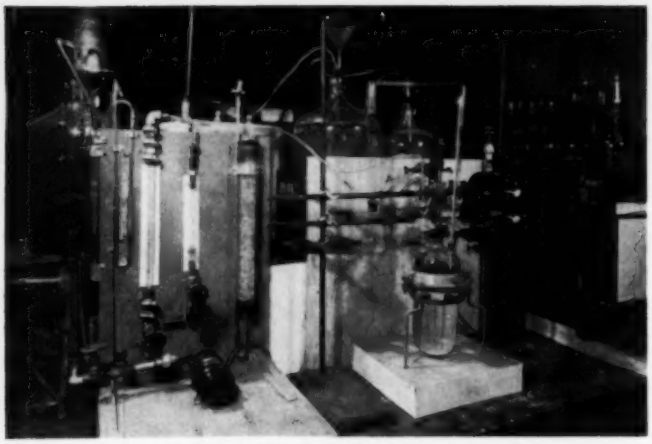

Fig. 2. Experimental apparatus.

Literature

Only a limited amount of literature is available to date on the subject of agitation in batch-type blending or mixing equipment or in continuous flow reactors (*2, 8, 9, 21, 25, 29, 30, 31, 34*). As a result, design engineers have been forced to depend almost solely upon the recommendations of mixing-equipment manufacturers.

Batch-Type Mixing. Mixing times in a homogeneous system were determined (*34*) by adding a small volume of brine solution to a wooden vessel filled with water, and measuring the time required for the concentration in the vessel, as indicated by four conductivity cells, to become uniform. The same type of experiment was conducted by Stead, Page and Denbigh (*30*) who measured mixing time by observing the time required for disappearance of color from a starch-iodine solution after introducing a drop of thiosulfate solution. Dodd (*8*) used a photometric method, dependent on refractive index, for measuring mixing times for mixtures of hydrocarbons, but used a reciprocating wire gauze mixer so that his data are difficult to apply. A mathematical approach has been presented (*4*) which shows that intimacy of mixing varies exponentially with time, but does not relate mixing at different intensities of agitation. An approximate empirical equation for mixing time under various conditions was given by Serner (*29*), who also points out that mixing time depends on efficiency in the use of power input as well as on the power absorbed. Valentine and MacLean (*31*) list the preferred types of mixing equipment with power requirements for many operations, including simple mixing.

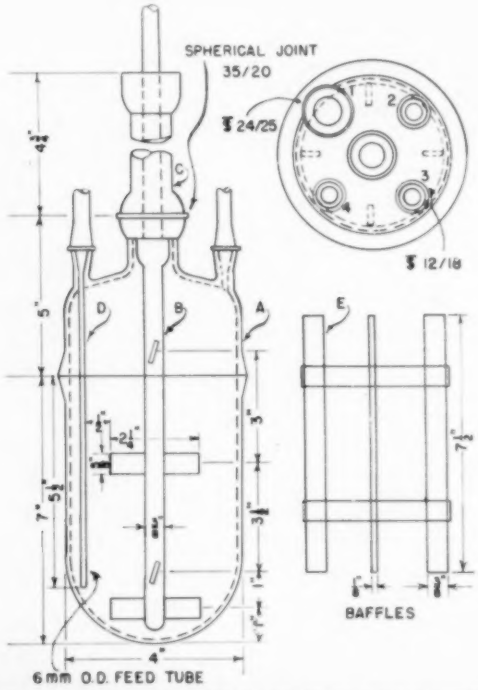

Fig. 3. Diagram of experimental continuous reaction vessel.

Continuous Blending. A method of determining blender efficiency was suggested (*2*) for the case where batches of varying concentration are fed into a stirred tank in order to produce a more uniform product. This analysis does not apply to the case where a reaction takes place in the vessel. Experimental verification of continuous feed stirred tank reactor theory in laboratory equipment has been made (*9, 21, 25, 30*) showing that essentially perfect mixing was attained at agitator speeds of 80, 325, 210 and 3000 rev./min., respectively.

Mixing Times

Mixing time, t_m, is here defined as the time required for a slug of feed solution to be dispersed homogeneously throughout a vessel in a batch operation or in a continuous flow system. With low intensities of agitation the mixing time will be large, while if agitation is increased sufficiently the mixing of an entering material or stream takes place almost instantaneously and the mixing time approaches zero.

The mixing of mutually soluble liquids is a three-dimensional shuffling operation and is directly dependent upon turbulence in the vessel. For a continuous flow system, both agitator action and flow of feed into the vessel contribute to this turbulence and this in turn determines the mixing time.

A modified Reynolds number for agitation $(nd^2\rho)/\mu$, has been successfully used in correlating the turbulence caused by agitator action, with power consumption, (*3, 14, 17, 22, 23, 25-27, 33*), heat-transfer coefficients (*6, 12, 28*),

and mass-transfer coefficients (*11-13, 15*), in agitated vessels. It is probable that mixing times can be correlated in a like way with agitator speed and vessel design by using a modified Reynolds number.

The intensity of mixing caused by introduction of a feed stream into a vessel has not been previously studied as far as known.† By measuring mixing time in a vessel as a function of both feed rate and agitator speed, it will be shown that their independent and their combined effects can be measured and correlated. The measurement of mixing time appears to be extremely useful since it gives a direct measure of the intensity of agitation within a vessel, independent of the agency causing that agitation.

Mixing Time Measurements. Measurements of the effect of agitation intensity on mixing time (using a dye), and on the amount of chemical reaction obtained were carried out in a glass laboratory stirred tank reactor fitted with a continuous flow feed system (Fig. 2, 3). This feed system supplied a measured stream of distilled water for the mixing-time experiments or a measured mixture of partially reacted acetic anhydride and water for the reactor performance experiments. A 1¾-in. diameter dissolving column, 20 in. long, and packed with ¼-in. lengths of 4-mm. glass tubing, provides solution and mixing of the acetic anhydride in the water. The resultant homogeneous solution is run into a single 1950 cc. Ace Glass high pressure Pyrex autoclave (Fig. 3). For measurement of reactor regulation the vessel was immersed in a thermostated bath which maintained constant vessel temperature (±.05° C.) even at low agitation intensities. The paddle agitator, *B*, guided by the ground glass bearing, *C*, directs the liquid downward, and was driven through a belt system by a Cenco variable speed stirrer. Baffles, *E*, are placed inside the vessel, *A*. Reaction solution entering through neck 4 is led to the bottom of the reactor by the 3.5-mm. I.D. feed tube, *D*, and leaves at the top of the vessel through neck 2.

Measurement of mixing time was accomplished by observing the time required for the homogeneous distribution throughout the glass vessel of a small volume (3 cc.) of dye solution entering with the feed stream. This dye was injected through a three-way cock into the continuously flowing feed stream of water, and was dispersed into the water-filled vessel. The temperature of the system was maintained by the introduction of feed water at a controlled temperature between 20° C. and 30° C.

The dye was introduced within a time less than 15% of the total mixing time, and at a sufficiently low rate so as to eliminate the possibility of density differences interfering with mixing. When the dye is introduced slowly, the dye solution entering the reactor is well diluted by the water feed stream and has nearly the same density as water. This dilute dye can therefore be expected to follow the same motions as the surrounding water and the mixing time for the dye becomes the same as mixing time for any other slug of feed. If the dye is introduced at the vessel bottom in too great a concentration, a pool of the more dense dye solution may form there, considerably increasing the time for mixing. Density of the saturated dye (Ponceau *G*) solution is 1.089 g./cc., compared with 0.998 g./cc. for water at room temperature.

Mixing time, t_m, is recorded as the time required, after first injecting the dye, for the dye to become distributed throughout the vessel and all visible variations in color density to disappear. When the dye solution appeared in the vessel effluent before all variations in color within the vessel had disappeared, the time for complete disappearance of the variations in color density was taken as the time for complete mixing. That this procedure was satisfactory and reproducible is shown by the agreement of the data in Figure 6. The progressive stages of mixing of the dye are shown in four photographs of Figure 4.

Figure 5 (Table 1) shows the observed effects on mixing time, t_m, of agitator speed, n, and of the feed rate, F. The latter factor has been plotted as the vessel holding time θ which equals V/F where V is the volume of the vessel and F is the volumetric feed rate. This plot shows that increasing agi-

† MacDonald and Piret know of no such study.

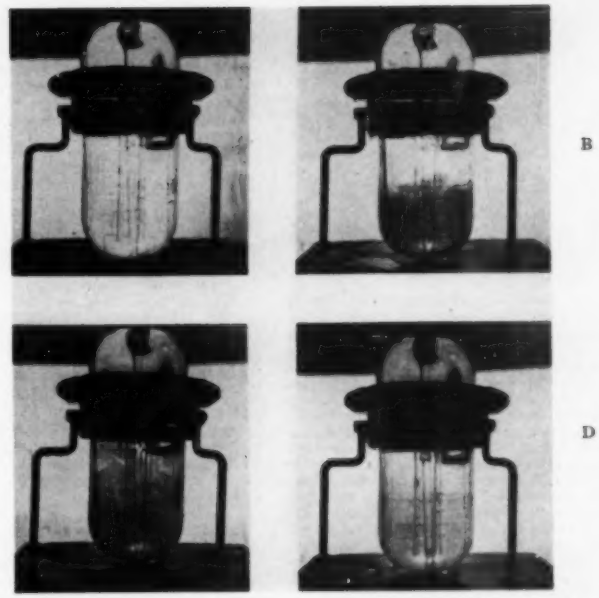

Fig. 4. Progressive stages in mixing a slug of dyed feed throughout a continuous feed stirred tank. Dye enters near bottom and side of vessel through a tube at the extreme left side of the vessel. (Agitator speed 10 rev./min. and holding time 25 min.)

A. Vessel filled with water only ($t = 0$ min.).

B. Dye just introduced at vessel bottom ($t = ¼$ min.).

C. Dye spreading throughout the vessel ($t = 2$ min.).

D. Complete homogeneity within the vessel ($t = 4$ min.).

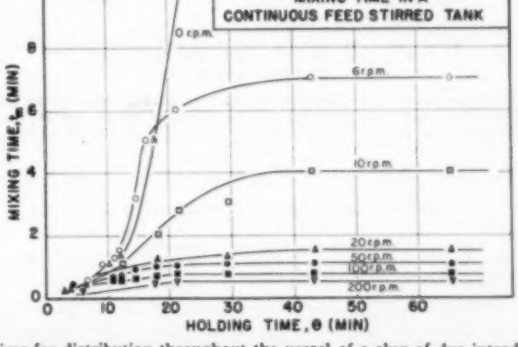

Fig. 5. Time for distribution throughout the vessel of a slug of dye introduced into the feed stream, given as a function of agitator speed and holding time (Table 1).

TABLE 1.—MIXING TIMES

Measured by Dye Dispersion in Glass Mixing Vessel (1950 cc.)

Run No.	Feed Stream Flow Rate F	Holding Time θ	Agitator Speed n	Mixing Time t_m
	cc./min.	min.	rev./min.	min.
26	67	29.1	0	15.
27	67	29.1	6	6.
28	67	29.1	10	3.
29	67	29.1	20	1.25
30	67	29.1	50	1.0
31	67	29.1	100	0.75

tator speed or decreasing vessel holding time (increasing feed rate) results in decreasing mixing time. A study of the curve (Fig. 5) for a single agitator speed, say 10 rev./min., will most readily permit an understanding of the effect on mixing times of the experimental variables. At holding times greater than 40 min. the contribution of agitator action is controlling, and it is seen that a change in feed rate has little effect upon mixing time. In the region between holding times of 40 min. and 5 min., the contribution of feed stream energy to agitation becomes increasingly important; and finally below a holding time of 5 min., the contribution of feed stream energy is controlling, and, as shown by the convergence of the several curves, changes in agitator speed have little effect.

The mixing-time data as replotted in Figure 6 resemble other published correlations for such agitation effects as heat transfer, mass transfer, and power requirements (*3, 6, 11-15, 17, 22, 23, 25-28, 33*). A definite break in the plotted data, at about 20 rev./min., corresponds to a critical Reynolds number $(nd^2\rho)/\mu$ of 10,400, which falls within the wide range of similar breaks found in the just-mentioned correlations. This value of Reynolds Number corresponds to a peripheral agitator speed of 0.2 ft./sec. A more extensive study in which liquid viscosity and agitator size are varied would be necessary to demonstrate the usefulness of the more general Reynolds number in this correlation.

In measuring mixing times by observing dispersion of dye, a change in liquid flow patterns within the vessel was observed at agitator speeds of about 20 rev./min. At agitator speed below 20 rev./min., the dye solution is gradually mixed throughout the vessel, but the region within and near the rotational plane of the paddle becomes homogeneous much sooner than the bulk of the liquid. Apparently, vertical mixing at these low speeds occurs mainly as a result of eddy currents formed around the moving paddles. Above about 20 rev./min., however, centrifugal action becomes more important and the liquid in the region of the paddles is forced radially outward against the vessel wall and then vertically into the bulk of the liquid. Thus viscosity limits mixing at low speeds, while inertia effects become limiting above theoretical Reynolds number. These observations agree with the statement of Mack and Uhl (*23*) that above the critical region centrifugal pump action occurs.

Effect of Feed Stream Action on Mixing Time. It is interesting that the data represented in Figures 5 and 6 can be used also to compare the effectiveness of agitation caused by the feed stream vs. that caused by the agitator.

From the mixing-time measurements of Table 1, values are chosen for agitator speed alone at zero feed rate, and for feed rate alone at zero agitator speed which result in equal mixing times. These values of agitator speed and feed rate for several mixing times are given in Table 2. Power consumption at each agitator speed has been calculated by the equation of Hooker (*17*) for geometrically dissimilar agitators, and the power consumption of the feed stream forced into the vessel has been calculated on the basis that all the kinetic energy of the feed stream is used in mixing. This latter assumption is, of course, arbitrary and serves only as a basis for our purpose. It was found experimentally that nearly equal values of power input result from pressure-drop measurements across the vessel used and from kinetic energy calculations.

To relate the contributions of agitator action and feed stream action, a relative efficiency factor, E, has been used. This factor is defined as the ratio, expressed in per cent, of the power used by the feed stream to the power used by agitator action in producing equal mixing times (Table 2). This relative agitator efficiency is plotted in Figure 7 against the agitator revolutions per minute. It can be seen that as the agitator speed is increased the energy input to the agitator becomes less and less effective when compared to the energy introduced by the feed stream.

The "total effective power input" for any combination of feed stream and agitator effects was then calculated by adding the feed stream power input to the product of the agitator power consumption and the efficiency factor. All the mixing-time data obtained (Table 1), vs. the total effective power input calculated as above are shown in Figure 8. Data for agitator speeds below 20 rev./min. are not included.

It appears from Figure 8 that on the basis of the present data, the same mixing time is obtained for any particular value of total effective power input, regardless of which combination of feed rate and agitator speed had been used. As is the case in other agitator studies, the specific data obtained can be expected to depend on such factors as baffles, vessel and agitator geometry, position and direction of feed and outlet openings.

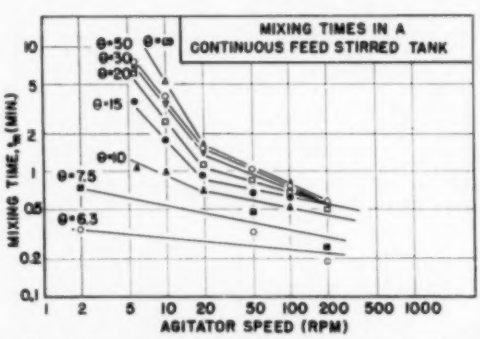

Fig. 6. Logarithmic plot of time for distribution throughout the vessel of a slug of dye introduced into the feed stream vs. agitator speed, for different holding times. Critical flow region (20 rev./min.) occurs at Reynolds number of 10,400 (Table 1).

TABLE 2.—CALCULATION OF AGITATOR EFFICIENCY

Mixing Time (t_m) (min.)	Agitator Speed (n) at ($\theta=\infty$)	Feed Rate (F) (cc./min.) at ($n=0$)	Holding Time (θ) (min.)	Calculated Agitator Power (hp./gal.)	Calculated Feed Stream Power (hp./gal.)	Relative Agitator Efficiency E %
½	200	300	6.5	7.04×10^{-4}	$.035 \times 10^{-4}$	0.50
⅝	150	260	7.5	2.97	.023	0.78
¾	100	230	8.5	.88	.016	1.82
1	50	190	10.3	.110	.009	8.30
2	20	150	13.0	.007	.0044	62.5
5	10	110	17.7	....	.0017	.....

Reactor Performance and Mixing Time

The effect of mixing intensity upon the behavior of a continuous feed stirred tank reactor was studied by measuring the degree of completion, D_e, experimentally attained by a chemical reaction occurring in the system.

$$D_e = 1 - C_1/C_0 \qquad (1)$$

where

C_0 = concentration of reactant in feed to reactor

C_1 = concentration of reactant in effluent from reactor

This value of D_e was then compared with the theoretical degree of completion, D_t, for a first-order reaction, based upon the assumption of perfect mixing in the reactor (*9, 24*).

$$D_t = 1 - \frac{1}{1 + k\theta} \qquad (2)$$

where

k = reaction rate constant

θ = nominal holding time of vessel

The hydrolysis of acetic anhydride, a first-order reaction, was used in this work. It was found experimentally that, at low intensities of agitation, the observed values of D_e fluctuate in an unpredictable way, sometimes being above and sometimes be-

low the theoretical value, D_t. At times, D_s would remain steady and either above or below D_t for periods of hours, and at other times, D_s would vary erratically, changing by as much as 5% to 10% within a few minutes. The value of D_s measured at any given time depends upon the actual pattern of flow which is set up within the reactor. By observing the flow of dyes, this pattern was found to vary considerably, over a period of time, even though the operating variables apparently remained constant.

The behavior of the system was then correlated in terms of the "regulation" of the individual values of D_s observed at different times; that is, in terms of the percentage deviation of the experimental degree of completion from the theoretical value. The per cent regulation for the reactor system is here defined as

$$\text{Per cent regulation} = \frac{D_s - D_t}{D_t} \times 100 \qquad (3)$$

where D_s is the value for experimental degree of completion at the time of measurement, and D_t is the theoretical degree of completion based upon perfect mixing in the reactor. Per cent regulation may also be expressed in terms of the concentrations entering and leaving the reactor.

$$\text{Per cent regulation} = \frac{C_{1,t} - C_{1,e}}{C_o - C_{1,t}} \times 100 \qquad (4)$$

where

$C_{1,e}$ = experimental concentration of reactant in effluent
$C_{1,t}$ = theoretical concentration of reactant in effluent
C_o = concentration of reactant in vessel feed

Thus, as the individual measured values of D_s vary above and below the theoretical value D_t, the individual values for regulation vary above and below zero. For high intensities of agitation it has been shown experimentally (*9, 21*) that D_s is constant and agrees with D_t within the experimental error. In these cases the regulation approaches zero.

The object of studying reactor behavior at low agitation intensities was, in part, to find experimentally the effect of insufficient agitation on the effluent concentration and also to investigate the nature of the relation between mixing time and regulation. It seems apparent that as holding time in the vessel is decreased, a given finite time for mixing of the feed will have an increasing effect upon the degree of completion attained. The fraction of the holding time required for mixing, that is t_m/θ, was therefore considered to be a more significant term than mixing time alone in affecting the performance of a continuous flow reactor. As the function t_m/θ is reduced and approaches zero, the limits of regulation should likewise decrease and approach zero, indicating that perfect mixing is more nearly attained.

To test this postulate, the experimental degrees of completion, D_s, were measured for the continuous reaction of acetic anhydride and water in the single Ace Glass high pressure autoclave system (Figs. 2, 3, and Table 3). The system is operated at constant temperature and flow rates for a period of at least seven times the holding time, θ, before taking samples, to insure that the original constants have been purged and steady-state concentrations obtained in all reactors (*18, 20*). The analytical method of Caudri (*5*) and Vles (*32*) was used to determine the steady-state concentrations. To check these measurements and to supply additional data, use was also made of the results obtained by Eldridge (*10*). His system was composed of a series of five vessels whose design was identical with that of the single vessel used in this work (*9*).

Figure 9 illustrates the effect of imperfect mixing, which is expressed here in terms of the ratio, t_m/θ, upon the limits of regulation of the reactor system. For this purpose the mixing time, t_m, was found from Figure 5. It may be seen (Fig. 9) that at each value of the mixing time parameter, t_m/θ, the individual values of regulation fall within a range extending about equal in number and magnitude above and below zero. The extreme limits of this range, as well as the average deviations of the values from zero, apparently depend upon the value of t_m/θ employed. As t_m/θ is decreased, indicating more perfect mixing, the limits of regulation also tend to decrease, showing closer agreement between D_s and D_t. It is to be noted that even at the lowest intensities of agitation the values for regulation did not exceed 20%. Thus even at the lowest intensities of agitation studied ($\theta = 27$ min., $n = 0$ rev./min.), the measured values of D_s agree at least roughly with D_t. Reaction systems in which large variations in densities or viscosities occur can be expected to show quite different characteristics.

Because the regulation appears to approach zero asymptotically, it is proposed that a satisfactory intensity of agitation for a stirred tank reactor be considered as that necessary to maintain the fluctuations in effluent concentration within the limits imposed by subsequent operations or product requirements.

Flow Patterns in the Reactor. Experiments were also performed to determine the pattern of flow within the reactor which caused the previously observed fluctuations in effluent concentration at low intensities of agitation. In these experiments a small volume of dye solution was introduced into the vessel feed stream after the concentration

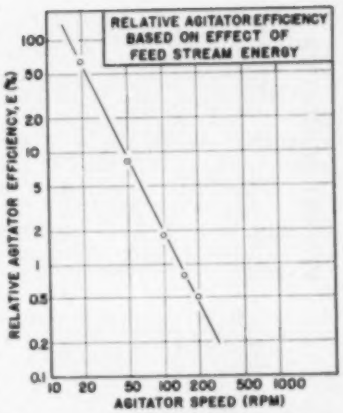

Fig. 7. Relative efficiency of agitator power as a function of agitator speed.

measurements had been made for some of the reaction runs of Table 3. Visual observations were then made of the flow of this dyed feed in the vessel.

At low intensities of agitation if the incoming reactant solution is less dense than the product solution, a channel tends to form in the reactor between the feed opening near the bottom and the exit at the top of the reactor. This phenomenon occurred during the reaction of acetic anhydride and water (Fig. 10, *A, B, C*). Here the density of the reactant anhydride solution (0.18 *N*) is 0.9986 at 25° C. and that of the product acetic acid solution (0.18 *N*) is 0.9988 at 25° C. This phenomenon was used by Kilpatrick (*19*) in reaction rate studies and was confirmed for this study by direct observations. It

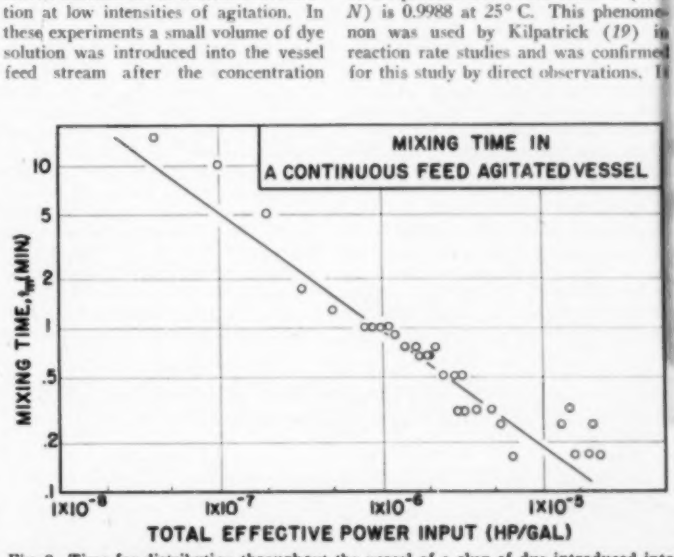

Fig. 8. Time for distribution throughout the vessel of a slug of dye introduced into the feed stream given as a function of "total effective power input" resulting from agitator action and feed stream action.

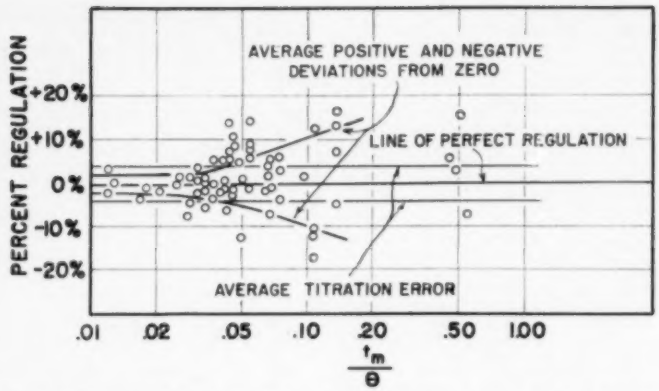

Fig. 9. Reactor regulation vs. t_m/θ for the continuous reaction of acetic anhydride and water, indicating increased limits of regulation for decreasing agitation (Table 3).

the entering feed solution is more dense than the reactor contents, as for example when a feed of acetic anhydride solution is pumped into a reactor filled with distilled water, a quite sharply defined separation may occur. A flow pattern is obtained (Fig. 10*D*) in which little vertical mixing occurs and this condition approximates that of a tubular-type reactor.

Effect of Channeling on Degree of Completion. Channeling occurs when the entering feed stream does not mix with the entire contents of the vessel but follows a channel of lesser volume or diameter than that of the entire vessel. Furthermore, in these channels longitudinal mixing is poor and the flow resembles that of a tubular reactor. Although with increasing levels of agitation distinct channels are not observable, the flow pattern can still be described as channeling where the size and position of the channels fluctuate rapidly with time.

Channeling in the reactor can, depending on the effective diameter of the channel, cause the degree of completion of the reaction to be above or below the value predicted for perfect mixing. When a channel of small effective diameter is formed between the vessel inlet and outlet, the degree of completion attained approaches that for a tubular reactor having a smaller volume than the vessel being used. The remaining volume is essentially dead volume containing completely reacted solution which is retained for indeterminate periods within the vessel. The calculated degree of completion for a reaction decreases as the effective reaction volume decreases, and for a channel of small volume it will be less than the degree of completion based upon perfect mixing throughout the entire reactor. If the channel increases in diameter and volume to nearly that of the entire vessel, the degree of completion attained will be greater than that for a vessel with perfect mixing. It should be remembered that except for zero-order reactions, a greater degree of completion is always attained in an ideal tubular reactor than in an ideal stirred tank reactor of equal volume (*7*).

Over periods of time varying from a few seconds to say ten or fifteen minutes, the colored channel formed in the reactor was observed to fluctuate considerably in an erratic manner and without apparent cause. As fluctuations in channel size and position occur, it is possible for the measured degree of completion to vary not only between the limits indicated in the preceding paragraphs for steady operating, but also between a value near zero for a slug of unreacted solution and a value of unity for a slug of completely reacted solution coming from the "dead volume" previously mentioned. However, as recorded in Table 3, only fluctuations up to ±20% from the theoretical were observed.

In some of the runs recorded in Table 3, the effluent concentration was examined continuously by placing two platinized platinum wires in a 0.5-cc. cell and causing the product stream to flow directly through this cell. The conductivity was measured with a bridge circuit using an electric-eye vacuum tube to detect fluctuations. The eye opening was sensitive to changes of 1.0% in conductivity with only about 1/5-sec. time delay in readings. No rapid fluctuations in the conductivity, as shown by the steadiness of the electric eye, were observed under any conditions. However the effluent concentration was observed to change by perhaps 2% to 10% of the theoretical value, on samples taken at intervals of more than 15 min.

TABLE 3.—EXPERIMENTAL CONDITIONS AND MEASURED DEGREES OF COMPLETION

For the Acetic Anhydride-Water Reaction in a Single Continuous Feed Stirred Tank Reactor (1950 cc.)

Run	Agitator Speed (rev./min.)	Holding Time (min.)	Theoretical Value D_t	Measured Value D_e	Per Cent Regulation	Mixing Time from Table 1 min.	t_m/θ
1	0	26.0	0.750	0.795	+5.7	12.	0.461
2	35	26.0	0.750	0.795	+6.0	1.0	0.038
3	245	26.0	0.750	0.760	+1.3	0.75	0.029
4	0	27.1	0.757	0.701	−7.4	15.	0.553
5	35	27.1	0.757	0.822	+8.6	1.25	0.046
6	120	27.1	0.757	0.701	−7.4	0.75	0.028

Summary

When a chemical reaction is carried out in a continuous feed reactor with less than ideal mixing, the observed concentration of the effluent stream fluctuates both above and below the theoretical values and the magnitude of the fluctuations depends upon reactor design, characteristics of the solution in the reactor, and also probably upon the reaction rate. On increasing mixing intensity in the reactor, the fluctuations diminish and the outlet concentration approaches closely the theoretical value calculated on the assumption of perfect mixing in a continuous feed reactor.

The effectiveness of mixing in the reactor was expressed in terms of the mixing parameter: time required for mixing divided by the total reactor holding time. It was found necessary to express mixing intensity in terms of mixing time, in order to include the mixing effects of both the rotating stirrer and the incoming feed stream. The dependence of mixing time upon stirrer speed and feed stream rate was measured so that the characteristic mixing time would be known for any combinations of stirrer speed and feed rate used during reaction rate measurements.

During these studies, the existence of a critical stirrer speed was established, and a correlation was developed between mixing time and total effective mixing power, by defining the total effective mixing power as the feed stream power input added to the product of stirrer power consumption and relative stirrer efficiency.

Notation

C = molar concentration of reactant

d = length of agitator paddle

E = relative agitator efficiency (based on mixing effect of feed stream)

F = volumetric rate of flow

k = specific reaction velocity constant

n = agitator speed (rev./min.)

t_m = mixing time

V = vessel volume occupied by reaction medium

μ = liquid viscosity

ρ = liquid density

$\theta = V/F$ = nominal holding time of vessel

Acknowledgment

The assistance of the Engineering Experiment Station of the University of Minnesota in the preparation of this manuscript is gratefully acknowledged.

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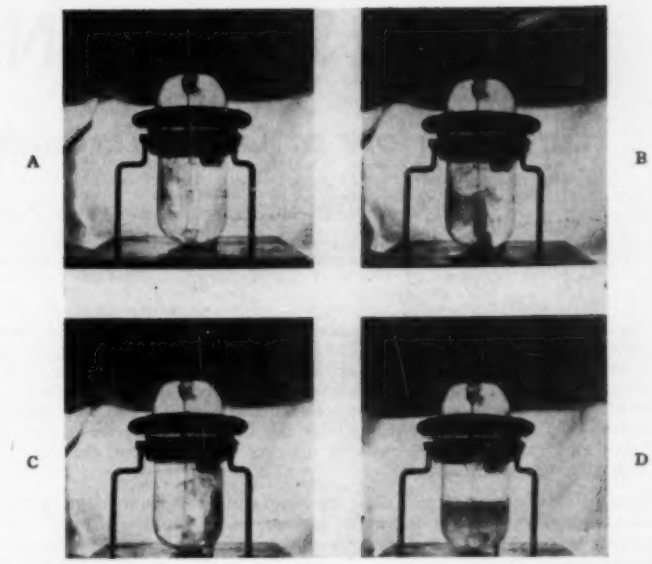

Fig. 10. Photographs showing flow through a reactor for different feed rates, giving channeling (A, B, C) for feed less dense than reaction products, and tubular reactor action (D) for feed more dense than reaction products. Dyed feed is shown entering the vessel through the feed tube at the lower left.

A. Channeling near feed tube (θ = 170 min., n = 0 rev./min.)

B. Channeling through central region (θ = 130 min., n = 0 rev./min.)

C. Channeling opposite feed tube (θ = 100 min., n = 0 rev./min.)

D. Boundary formation in tubular reactor (θ = 50 min., n = 0 rev./min.)

Discussion

C. R. Wilke (University of California, Berkeley, Calif.): Theoretically doesn't it take infinite time to achieve perfect mixing and do you have to make an arbitrary line of demarcation so that the accuracy of your analyses no longer indicates a finite change?

R. W. MacDonald: Theoretically an infinite time should be required for complete molecular redistribution within the reactor. However, when a portion of dye is injected into the vessel, mixing proceeds rather rapidly to the point where the contents of the reactor appear completely uniform and no further changes in appearance can be discerned. It is also of interest to note that this end point is quite reproducible even when observations are made by several different people.

(Presented at Forty-third Annual Meeting, Columbus, Ohio.)

MASS TRANSFER

Laminar Flow of Gases Through Granular Beds

MERK HOBSON † and GEORGE THODOS

Northwestern Technological Institute, Evanston, Illinois

The kinetics of physical mass transfer as applied to gases in single phase laminar flow through granular beds has been studied for a variety of binary systems. These studies include the vaporization of water, *n*-butanol, toluene, *n*-octane, and dodecane from the surfaces of spherical packings into air, nitrogen, carbon dioxide, and hydrogen. The experimental data have been correlated on the basis of the *j* mass-transfer factor and the modified Reynolds number applicable to the flow of fluids through granular beds.

Based upon existing correlations for single phase flow through porous beds, a critical modified Reynolds number of 50 has been found comparable to the 2100 Reynolds value above which laminar flow normally disappears in circular conduits. An analysis of the present experimental results in conjunction with values previously reported suggests that this critical modified Reynolds value is also applicable to the mass-transfer correlation for granular beds. A critical evaluation of the data below a modified Reynolds number of 50 indicates that a linear power distribution is applicable. For values of the modified Reynolds number above 150, a linear power distribution is equally applicable.

IN recent years the mass-transfer factor, as originally developed by Chilton and Colburn (*2*), has been used by numerous investigators as a dimensionless parameter for the correlation of experimental data in the flow of fluids through granular solids. Based upon experimental studies in the vaporization of water into air from the surfaces of spheres and cylinders, Gamson, Thodos and Hougen (*3*) proposed the mass-transfer factor

$$[j_d]_g = \frac{k_g p_{gf} M_m}{G} \left[\frac{\mu}{\rho D_v}\right]_f^{2/3}$$

as a generalized variable for the estimation of mass-transfer coefficients in single phase flow through granular beds.

A mass-transfer factor specifically applied to transfer across a liquid film

$$[j_d]_l = \frac{k_l c_{lf} M_m}{L} \left[\frac{\mu}{\rho D_l}\right]_f^{2/3}$$

was employed by McCune and Wilhelm (*8*) and Hobson and Thodos (*5*) to correlate studies in solid-to-liquid and liquid-to-liquid transfer respectively.

In both these investigations the transfer surfaces were spherical, facilitating a direct comparison with the original transfer factor correlation for gases. The correlations for both liquids and gases produce an identical function when correlated with a modified Reynolds number, $D_p G/\mu$, as the independent variable. This combined function has been advocated as a completely general method for the prediction of mass-transfer coefficients in the specific case of single phase flow of fluids through granular solids.

Reliable data for beds composed of spherical particles applicable to gas-film transfer on the gas film are restricted to the single system, air-water. To broaden the foundation for general acceptance of the correlation, additional experimental information for a diversity of gaseous systems would be advantageous. Furthermore, the data on the air-water system which are available involve the assumption of an adiabatic saturation temperature in the plane of vaporization. The validity of this assumption is open to question in the low range of flow rates corresponding to the range of modified Reynolds numbers covered by the liquid-transfer studies.

The portion of the curve established by the liquid-transfer data indicates the possibility of a transition point somewhat analogous to the transition from laminar to turbulent fluid flow. Further experimental study within this range of flow characteristics would be valuable for more accurate definition of the correlation in the low Reynolds region. A direct comparison between the transition from laminar flow in circular conduits at a Reynolds number of approximately 2100, and a similar transition for single phase flow through porous beds is possible through the contributions of Brownell, Dombrowski and Dickey (*1*). This evaluation becomes possible from a knowledge of the porosity and sphericity of the particles constituting the bed. From these characteristic properties, it is possible to predict an empirical factor, which, when multiplied by the modified Reynolds number for the packing, produces a Reynolds number equivalent to that for flow through circular conduits. For an average porosity of 0.412 applied to beds of spherical particles, a modified Reynolds number of 50 corresponds to the value of 2100 for circular conduits.

The present work attempts to strengthen the generality of the mass-transfer factor as applied to granular beds by extending a variety of gas-transfer systems into the range of Reynolds numbers covered by the liquid-film experiments while eliminating some of the questionable assumptions, which in the past have been applied in the interpretation of such data, specifically the use of the adiabatic saturation temperature in the plane of vaporization.

Such variables as temperature and particle size have been previously studied over a wide range and are not specifically considered in this treatment.

Experimental Procedure. The experimental technique applied to these studies represents a quantitative analysis of liquid vaporization rates from the available transfer surface in a bed of porous spheres. All experimental measurements were restricted to a predetermined constant rate period to insure transfer under steady-state conditions. Single phase flow of gases through the bed provided only gas-film resistance to mass transfer.

The equipment adapted to these experiments is indicated in Figure 1. The central section of the reactor, equipped with an internal screen support for the bed of spheres, served as a vaporization chamber. The inside diameter of the reactor was 2.06 in. The joints between the three sections of the reactor were machined and gasketed in such a way that the upper reactor section provided sufficient bearing

† Present address: University of Nebraska, Lincoln, Neb.

pressure to effect a tight seal at the low pressures used in the experimental work.

To facilitate rapid removal of the bed, the upper section was connected to the inlet piping by means of a flexible joint. Quick opening valves were installed in the gas feed line and into a by-pass connection immediately preceding the reactor for virtually instantaneous introduction or cut-off of the gas flow to the vaporization chamber. Gas flow was measured by an orifice installation upstream from these valves. A pressure tap manifold permitted the use of either a vertical manometer or an inclined draft gage. Gases were supplied from high pressure cylinders, or in the case of air, from the building air supply under pressure regulator control. Inlet and outlet temperatures of the flowing gas were obtained by permanent thermometer installations. Pressure taps above and below the bed provided absolute pressures and pressure-drop data for the reactor. The reactor outlet piping included a connection for wet test metering of the gas flow for standardization purposes or for the low flow rates where the use of the orifice installation would introduce appreciable error.

No attempt was made to adapt the equipment to adiabatic operation, since all the runs were essentially at room temperature. Thermocouples were introduced into the vaporization chamber and permanently attached to three porous spheres. This was accomplished by drilling through a major diameter and inserting the thermocouple through the sphere until the tip reached a point just below the opposite surface. The dimensions of the vaporization section made it impractical to include a larger number of thermocouples.

This procedure eliminated the necessity of assuming that vaporization takes place at the wet-bulb temperature. It is highly probable that at the low gas velocities realized in this investigation, the actual surface temperatures would deviate markedly from the wet-bulb readings. In general, wet- and dry-bulb thermometry cannot be regarded as valid at the gas velocities within the laminar range.

A satisfactory operating procedure was evolved as a result of a number of preliminary runs using a water-air transfer system within the range of modified Reynolds numbers for which accurate data are available. The total amount of mass transferred was found to be relatively small and in order to maintain a sufficiently high degree of precision, the liquid vaporized was best determined by weighing the bed at the beginning and end of each run rather than at specified time intervals during the progress of the run. To avoid the possibility of extending a run beyond the point of steady-state transfer, the length of the constant rate period for each experimental system was investigated as a function of the gas rate. The total transfer surface and the temperature of vaporization also affect the length of the constant rate period. Neither of these quantities was varied appreciably in actual practice since these variables were not specifically under investigation. Average values were used in the above determinations, but to provide a margin of safety against minor deviations from the average, the runs were restricted to a maximum of 75% of the predetermined constant rate period.

An initial period of operation was allowed for the system to reach steady state. The bed was then quickly removed, weighed, and returned to the reactor for the quantitative portion of the run. The differential weight loss of the bed accounted for the liquid transferred to the gas phase. Although the time required for initial weighing seldom exceeded 45 sec., a slight shift from steady-state conditions was unavoidable. The period required for return to equilibrium, however, was not significant in relation to the total time for the run.

The spheres permanently equipped with thermocouples were invariably distributed, so that readings of surface temperature in the plane of vaporization were available for both the top and the bottom of the bed. The position of these spheres in the cross section of the bed was varied at random throughout the series of experiments.

All the experimental runs were based upon a porous spherical packing of 0.370 in. diameter. The principal physical characteristics of the packing include a void volume of 47.5%, 34,304 spheres/cu.ft. and a total transfer area of 102.5 sq.ft./cu.ft. As has been previously noted, other investigations have conclusively proved that the correlation includes the effects of temperature and packing diameter. Hence, this investigation was restricted to atmospheric conditions and one packing diameter as a matter of convenience.

Experimental Results. The procedures of the previous section were applied to the vaporizations of the following liquids in the indicated gases:

1. water-air
2. normal butanol-air
3. normal butanol-nitrogen
4. toluene-air
5. normal octane-air
6. normal octane-nitrogen
7. normal butanol-carbon dioxide
8. toluene-carbon dioxide
9. normal butanol-hydrogen
10. normal dodecane-hydrogen

These systems were chosen as representative of various types of chemical compounds offering a wide range of physical properties. A second consideration which influenced this choice stems from the experimental necessity of restricting the rate of vaporization to a point where gases in laminar flow will not be saturated with respect to the transferable component. Unless these effluent gases are below the point of saturation, the driving force for mass transfer cannot be properly expressed in the basic rate equation over the entire bed. In general, the liquids chosen were restricted to those possessing a relatively low volatility. From a practical standpoint, the available transfer surface and the mass flow rate of the gases were varied to avoid complete saturation. A bed depth of ¾ in., however, was considered to be a minimum for retention of the flow characteristics of a bed. This minimum bed depth in turn established the lower limit of gas flow below which the transfer data are not adaptable to rate calculations.

Experimental data for each of the above systems are summarized in Table 1. With reference to the surface temperatures, it will be noted that a variation in temperature exists at the top and the bottom of the bed. This may be due in part to the nonadiabatic nature of the vaporization and to the behavior characterized by gases in laminar flow. These surface temperatures represent actual physical measurements, and when used in conjunction with the rate of mass transfer, they permit an evaluation of the driving force applicable to the rate equation. The third thermocouple was used for comparative purposes to establish the nature of the surface temperature variation within the bed, both in the direction of and perpendicular to the gas flow. The variation in the direction of gas flow appeared to be approximately linear. Small variations in surface temperature were noted in the radial direction and as partial compensation for this variation, the thermocouples in the top and bottom of the bed were positioned at random over the entire series of runs.

Table 2 summarizes the calculated results including the mass-transfer coefficient and mass-transfer factor for each run. The

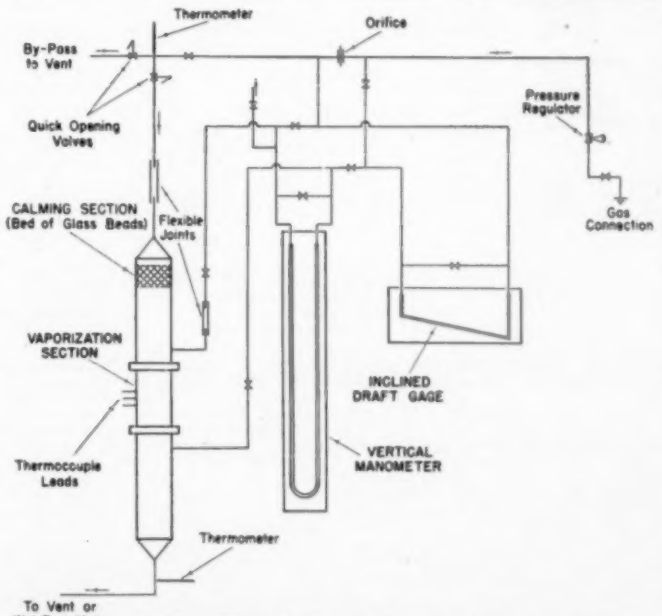

Fig. 1. Schematic diagram of vaporization equipment.

TABLE 1.—SUMMARY OF EXPERIMENTAL RESULTS

Run No.	Duration of Run Hours	Rate of Liquid Transfer lb./hr.	No. of Spheres In Bed	Pressures, mm		Temperatures, °F				Gas Flow SCFH 0°C.- 760 mm.	Inlet Wet Bulb Temperature °F
						Gas		Surface			
				Inlet	Outlet	Inlet	Outlet	Top	Bottom		
n-Butanol-Nitrogen											
BN-1	0.5833	1.236	65	751.4	751.4	73.2	68.1	68.6	63.6	14.3	
BN-2	0.6833	0.794	55	750.8	750.8	75.0	69.2	68.0	63.8	8.65	
BN-3	0.4167	1.503	55	755.3	755.3	76.5	73.0	70.6	65.5	17.9	
BN-4	0.5167	1.114	60	751.2	751.2	74.5	69.8	68.4	63.4	12.6	
n-Butanol-Air											
BA-1	0.5167	2.496	69	754.8	754.2	73.5	67.5	65.5	65.0	43.2	
BA-2	0.1333	5.359	76	747.0	746.5	76.5	72.0	71.4	68.3	81.5	
BA-3	0.2667	2.455	75	742.0	742.0	77.1	74.0	69.5	66.3	26.7	
BA-4	0.1375	4.922	79	751.8	751.8	78.5	74.9	71.1	67.3	66.0	
BA-5	0.1000	4.850	85	751.2	751.2	78.7	74.8	70.4	68.5	50.1	
BA-6	0.1000	3.020	65	750.2	750.2	78.8	75.7	71.8	69.2	34.5	
Toluene-Air											
TA-1	0.0417	11.99	80	745.3	745.3	78.5	75	58.5	53.5	29.0	
TA-2	0.0347	20.32	117	742.4	742.4	80.2	76	56.2	56.2	48.3	
TA-3	0.0445	27.43	131	742.5	742.5	80.9	73.0	59.1	52.5	65.5	
TA-4	0.0583	6.20	68	733.7	733.7	73	70.3	55.0	57.5	12.5	
TA-5	0.0500	9.52	83	733.8	733.8	72.4	69.8	57.5	53.5	22.5	
TA-6	0.0583	8.73	70	742.5	742.5	79.5	75.5	59.5	55.5	18.2	
n-Octane-Air											
OA-1	0.1000	7.077	63	743.1	743.1	81.5	77.1	67.2	64.5	28.2	
OA-2	0.1333	4.862	73	743.8	743.8	80.2	77.1	66.4	64.5	15.5	
OA-3	0.1500	3.763	73	743.9	743.9	77.6	74.5	66.7	64.9	10.4	
OA-4	0.0833	10.660	72	744.0	744.0	79.9	74.6	67.2	62.7	54.1	
n-Octane-Nitrogen											
ON-1	0.2167	4.965	70	749.6	749.6	78.3	75.0	69.5	63.8	16.1	
ON-2	0.2000	4.178	69	749.9	749.9	78.8	75.1	70.4	63.1	14.1	
ON-3	0.1083	7.756	92	751.2	751.2	79.8	74.8	70.8	64.3	20.3	
Toluene-Carbon Dioxide											
TC-1	0.2750	3.856	60	740.0	740.0	78.4	73.3	63.4	59.5	7.84	
TC-2	0.2542	4.848	72	740.0	740.0	78.8	74.8	65.0	57.1	10.1	
TC-3	0.3333	3.393	72	741.0	741.0	76.5	73.0	62.6	59.8	6.10	
TC-4	0.1667	5.912	82	741.5	741.5	74.5	68.0	59.8	54.9	13.3	
n-Butanol-Carbon Dioxide											
BC-1	0.3500	0.945	62	755.3	755.3	71.6	70.5	66.8	66.0	10.4	
BC-2	0.9500	1.311	112	754.1	754.1	72.3	70.0	67.3	65.0	13.1	
BC-3	0.6167	0.879	90	745.8	745.8	76.9	73.8	69.8	69.2	8.02	
BC-4	0.1667	1.627	73	745.5	745.5	77.8	75.3	70.6	69.3	16.8	
BC-5	0.2167	2.004	73	747.3	747.3	78.3	75.3	69.8	69.1	22.3	
n-Butanol-Hydrogen											
BH-1	0.0833	6.220	59	753.1	753.1	77.5	74.5	69.4	67.1	68.79	
BH-2	0.0167	5.608	63	752.9	752.9	78.2	75.0	69.8	66.8	63.67	
n-Dodecane-Hydrogen											
DH-1	0.6167	0.2314	115	753.9	753.9	81.1	81.1	81.1	81.1	22.87	
DH-2	0.4667	0.2565	115	736.1	736.1	81.5	81.4	81.5	81.5	27.56	
Water-Air											
WA-1	0.7790	1.877	62	765.4	763.3	78.5	65.4	54.5	52.9	51.5	54.0
WA-2	0.6917	1.393	57	761.0	759.9	77.7	69.5	61.6	55.0	32.0	54.6
WA-3	0.4512	5.370	100	763.9	761.5	77.3	64.0	55.5	54.0	135.0	53.2
WA-4	0.4380	4.047	100	756.5	755.9	77.7	61.3	52.5	51.5	107.5	53.0
WA-5	0.5295	3.273	75	754.0	753.6	78.6	64.4	55.5	54.0	84.5	53.0

Run Notation

BN = n-Butanol - Nitrogen	TC = Toluene - Carbon Dioxide
BA = n-Butanol - Air	BC = n-Butanol - Carbon Dioxide
TA = Toluene - Air	BH = n-Butanol - Hydrogen
OA = n-Octane - Air	DH = n-Dodecane - Hydrogen
ON = n-Octane - Nitrogen	WA = Water - Air

former was calculated from the basic rate equation applied to transfer across a gas film

$$r = k_p a V [\Delta p]_m$$

The partial pressure values for the transferable component at the interface were obtained from the vapor pressures presented in Table 3. With the exception of the vapor pressures listed for toluene, these data were taken from literature. Values for toluene resulted from an extension of the work of Willingham, et al (*11*) to the desired temperature range using the methods developed by Thodos (*10*).

A number of simplifying assumptions were possible without impairing the accuracy of the calculations. For systems in which air was used as the flowing gas, the calculated results indicated that the small amounts of water vapor could be considered as dry air in the evaluation of the physical properties of the system. The single exception to this practice was in the treatment of data with water as the transferable component. A second simplification seemed feasible as a result of the Schmidt group calculations for the normal butanol-nitrogen system. The evaluation of the film properties, viscosity, density, and diffusivity, for the various flow conditions produced only minor variations in the numerical value of this dimensionless group. This is a logical result, since average film temperatures remained essentially constant for all the experimental runs within a given system. Further, the generalized methods used to obtain the viscosity (*7*) and diffusivity (*4*) values are in themselves approximations. Therefore, the use of a constant Schmidt group for each system was considered to be in order with the over-all precision of the variables comprising this modulus. Average values of the Schmidt group appear in Table 2.

Conclusions

Mass-transfer factors have been obtained from the vaporization of water,

toluene, normal butanol, normal octane and normal dodecane into such gases as hydrogen, nitrogen, air and carbon dioxide supplementing the existing correlation based upon the vaporization of water in air. It will be noted from Table 2 that the Schmidt group varied from 0.61 for air-water to a maximum of 5.06 in the case of hydrogen-normal dodecane which represents approximately an eight-fold variation. It is interesting to note that for the air-water system, the measured temperature in the plane of vaporization deviates markedly from the wet-bulb temperature at the lowest gas flow rate, but approaches the wet-bulb temperature at increased rates.

In the present treatment, the modified Reynolds group, $D_p F/\mu$, has been used to facilitate a comparison with previous data in this field. This comparison is presented graphically in Figure 2. The present studies are in general agreement with the work of McCune and Wilhelm (*8*) and extend the results of Gamson, Thodos and Hougen (*3*).

A critical analysis of the data appearing in Figure 2 between modified Reynolds numbers of 20 and 100 was made in view of a possible transition from laminar flow as predicted by the correlations of Brownell, Dombrowski and Dickey (*1*) for flow through porous media. A critical value of a modified Reynolds number of 50 is comparable to the accepted 2100 value for flow through circular conduits based upon bed characteristics used in these studies. The majority of experimental points below the critical value are the result of liquid-film studies by Hobson and Thodos (*5*) on the systems isobutanol-water and methyl ethyl ketone-water. A critical review of the experimental technique emphasizes the reliability of the isobutanol-water data as opposed to the methyl ethyl ketone-water system.

It was found that the present data for a variety of gaseous systems for modified Reynolds numbers less than 50 when considered in conjunction with the more reliable isobutanol-water points, produced a linear power function with a negative slope of unity. This is in complete agreement with the established characteristics of laminar flow. The mathematical representation of this function for values of $\mathrm{Re} < 50$ is $j_d = 10/\mathrm{Re}$.

As might be expected, an exact definition of the correlation in the transition range is not readily apparent. At Reynolds numbers above 150, the correlation may be again represented by a power function, $j_d = 1.30/Re^{0.48}$. The recommended correlations are indicated graphically in Figure 2.

The present experimental work corroborates both the liquid- and gas-phase transfer data previously reported and permits more accurate definition of the

TABLE 2
SUMMARY OF CALCULATED RESULTS

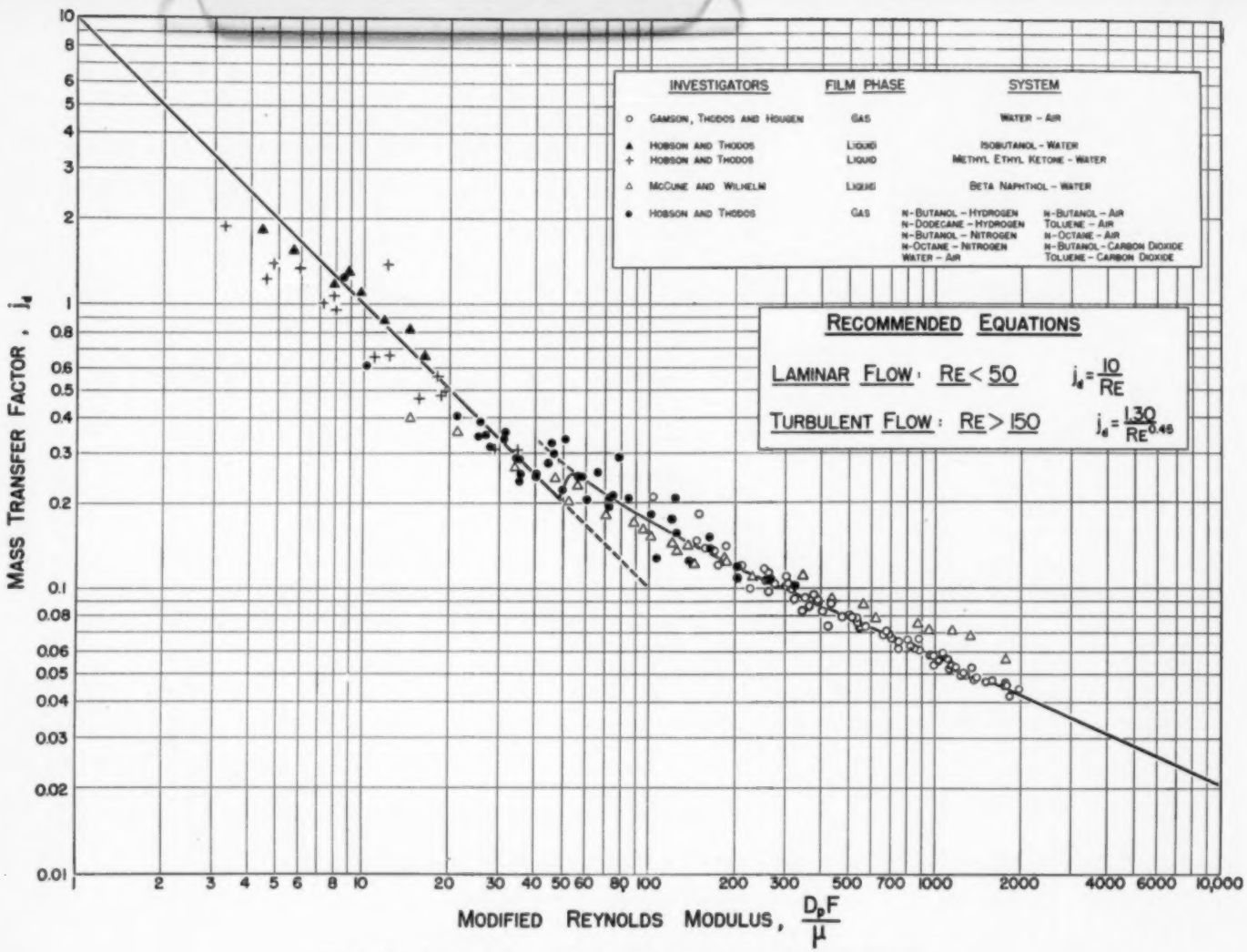

Fig. 2. Mass transfer factor—modified Reynolds modulus relationship for spheres.

correlation with the 8 to 300 range of values for the modified Reynolds number.

Acknowledgment

The authors wish to thank G. G. Lamb for his critical review of the manuscript and constructive suggestions in the analysis of the experimental results.

Notation

a = effective surface area of granular particles per unit volume, sq.ft./cu.ft.

c_{if} = mean concentration of nontransferable component, in the liquid film lb.moles/cu.ft.

D_l = diffusivity coefficient of transferable component in liquid state, sq.ft./hr.

D_v = diffusivity coefficient of transferable component in gaseous state, sq.ft./hr.

D_p = effective particle diameter, ft.

F = superficial mass velocity of flowing fluid, lb./(hr.)(sq. ft.)

G = superficial mass velocity of flowing gas, lb./(hr.)(sq. ft.)

$[j_d]_g$ = mass-transfer factor for gases

$[j_d]_l$ = mass-transfer factor for liquids

k_g = mass-transfer coefficient for gas film, lb.moles/(hr.)(sq. ft.)(atm.)

k_l = mass-transfer coefficient for liquid film lb.moles/(hr.)(sq. ft.)lb.mole/cu.ft.

L = superficial mass velocity of flowing liquid, lb./(hr.)(sq. ft.)

M_m = mean molecular weight of flowing medium, lb./mole

p = partial pressure of transferable component in gas film, atm.

p_{gf} = mean partial pressure of the nontransferable component in gas film, atm.

r = rate of mass transfer, lb.moles/hr.

V = volume of granular bed, cu.ft.

GREEK:

Δ = symbol for difference

μ = absolute viscosity, lb./(hr.)(ft.)

ρ = density, lb./cu.ft.

SUBSCRIPTS:

f = film

m = mean

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Discussion

John L. Dewey (Battelle Memorial Institute, Columbus, Ohio): Could you give some explanation of the rather large spread of the data at low Reynolds number, with this correlation?

Merk Hobson: I think that mass-transfer data, in general, have a tendency to spread. We have found that to be true in the past. In the case of the liquid data we are dealing with unsteady state transfer which involves extrapolation back to zero time. This undoubtedly is a factor in connection with the spread of these liquid data. In connection with the gas-transfer material, we had to depend upon a calculated material balance. If we had been able to check it experimentally, the results, perhaps, would have been in a little better agreement with the over-all curve that we finally established.

TABLE 3.—VAPOR PRESSURES OF LIQUID COMPONENTS

Hg. (mm.)

Temperature, °F.	Liquids				
	n-Butanol (6)	n-Dodecane (10)	n-Octane (10)	Toluene	Water (9)
40				8.94	6.29
50		0.0346	5.94	12.48	9.21
55		0.0451	7.07	14.65	11.07
60	3.49	0.0584	8.38	17.14	13.26
65	3.95	0.0751	9.90	19.98	15.80
70	4.71	0.0959	11.65	23.21	18.78
75	5.87	0.1218	13.66	26.88	22.23
80	7.42	0.1539	15.94	31.02	26.22
82	8.11	0.1681			

R. D. Morse (Du Pont Co., Wilmington, Del.): On your chart of Reynolds number vs. J factor, you showed two straight lines meeting at a transition region with a jog connecting those two lines, which looks similar to the meeting point for ordinary pipe line flow. I wonder if it is perhaps too much to expect that in a bed of granular materials where we do not have a homogeneous geometry we would have two straight lines and a narrow point of meeting where the jog would apply?

Merk Hobson: We certainly didn't mean to infer that they met. You will notice that the equation in the laminar region applies to a number of particles with a modified Reynolds number of 50. We would not express the correlation in terms of a power function again until we reach 150. Hence there is that differential between the 50 point and the 150 point, where I think the correlation is best expressed by a graphical average such as we have used. We indicated that region in dash lines. I think we have to use our own judgment in interpreting that particular region for the correlation. Certainly the equation does not apply.

R. D. Morse: Did you succeed in determining whether there is any entrance and exit effect for the rather shallow beds of large particles in small tubes you used?

Merk Hobson: No, we did not.

R. G. Taecker (Kansas State College, Manhattan, Kan.): How were surface temperatures obtained for the vaporization of these various liquids into gas streams?

Merk Hobson: The exact technique consisted of drilling through a major diameter of three of the spheres, and permanently installing fine wire thermocouples on the opposite surface. Now there may be some question as to whether the tips were indicating the actual interfacial temperature. I think we approached that about as closely as one can, experimentally, however.

R. G. Taecker: How were radiation errors minimized?

Merk Hobson: No attempt was made to make this operation adiabatic. We were not making any assumptions, as I said, with regard to adiabatic saturation temperatures. We compensated for this, however, by making the temperature of the runs all essentially room temperature. This was justified, since the correlation inherently includes temperature effects, a point that has been conclusively proved prior to our work. Hence temperature was not a variable under investigation.

(Presented at Kansas City (Mo.) Meeting.)

POLLUTION ABATEMENT AND INDUSTRIAL WATER MANAGEMENT

CHARLES F. HAUCK

Hall Laboratories, Inc., Pittsburgh, Pennsylvania

THE diminishing availability of water, coupled with increasing costs of treatment for usage and disposal have greatly magnified its importance in industrial plant planning and operation. As with taxes, some water costs are apparent, others are hidden—in power and fuel bills, equipment maintenance and replacement costs, etc. For this reason, plant-water problems should be dealt with expertly and carefully. In so doing, the many interrelationships among water-procurement-treatment-usage-disposal problems must be fully explored and exploited if lowest over-all plant-water costs are to be realized. This is the objective of the art and practice of industrial water management.

Abatement of pollution from waterborne industrial wastes constitutes a major problem of industrial water management. In many chemical and petroleum plants it is more difficult and expensive to condition water for disposal than for usage. This paper presents, for industrial plants, a stepwise program which will lead to solution of industrial waste problems for lowest over-all costs. The importance of simplifying and minimizing pollution problems before deciding upon treatment procedures and the importance of interlocking relationships among the problems mentioned will be stressed.

That water problems should not be considered or solved piecemeal is illustrated by the following case study. A paperboard mill harassed by diminishing water supply was considering purchase of land for development of supplementary wells. A co-ordinated study of all plant water problems revealed:

1. That well output had diminished to the point where supply was dangerously little more than demand
2. That water wastage was occurring due to a condenser slime problem and improper control of pumpage and storage
3. That correction of the mill's waste disposal problem would drastically reduce fresh water requirements

An inexpensive well-cleaning program immediately and substantially increased well capacity. Effective slime control, and better synchronization of water pumpage and storage with demand eliminated wastage. Conservation of raw water was also achieved through reuse of effluent. By better water treatment and usage, a problem in procurement of water was solved. In this way a major investment in additional wells was avoided and a co-ordinated plan for pollution abatement with a view to increasing the mill's raw water reserves was evolved.

Water problems should be approached with a knowledge of water technology, understanding of quantity and quality requirements at all water-usage points, and an appreciation of the principles of engineering economy. All these are necessary to attack effectively an industrial waste-disposal problem.

Such an attack may be likened to a major military campaign and logically divides itself into four phases as follows:

1. Reconnaissance—fact-finding
2. Preliminary maneuvers — defining, simplifying and minimizing the waste-disposal problems
3. Frontal assault—working out treatment and disposal processes, design and construction of disposal facilities
4. Consolidation of gains — continued practice of good industrial water management

Reconnaissance

In general before water problems can be brought into clear focus, reliable and complete data must be available regarding water quantity and quality requirements at various points of usage. To formulate a pollution-abatement program before all pertinent data are known is as unsound as it would be to start a military campaign without a map of the target territory.

Process Conditions. Flow sheets showing water and materials balances for all operations involving water will highlight the foci of pollution and the interrelationships of waste disposal with other water problems.

Stream conditions above and below the plant should also be known especially in areas where effluent quality is determined by the stream's ability to assimilate pollution.

Gauging and Sampling. It is a matter of self-interest as well as self-defense for industry to know what substances enter, pass through, and are discharged from its plants by the water route. Development of a materials or water balance for an operation or for a plant involves problems of flow measurement, sampling, and analysis. The innumerable problems encountered in analyzing industrial wastes will not be considered (*5*) but there are certain basic factors in sampling.

Frequently gross sampling errors spoil the results of arduous analytical procedures carried out to obtain high accuracy in examining industrial waste waters. Often we strive to reduce errors in analysis from 0.2% to 0.15% and blithely ignore errors of 10 to 20% in sampling. Sampling errors may result from how the sample is taken, where it is collected, or the methods of compositing and may be cumulative or compensating. Too frequently due to hidden sampling errors false conclusions are derived about efficiencies of waste-producing or waste-treating operations. Usually both spot and composite samples should be included in the program. Catch samples are needed to establish the shape and amplitude of concentration fluctuations. But the total quantities of waste components can be determined more easily from composite samples.

Choice of the sampling location requires consideration of the process cycle, sometimes synchronization of sampling at several points, hydraulic suitability, etc., and the carrier into which wastes are discharged. In general, points of great turbulence in streams, where hydraulic conditions are best for the nonseparation of solids, are most desirable for sampling. Even where the contaminants are soluble, homogeneity of samples is not assured.

Dissolved substances such as acids, bases, brines, dyes, etc., travel for great distances as slugs without blending with surrounding water. This is particularly true in large pipes, channels, rivers, etc. For example the north and west branches of the Susquehanna River flow as one stream for 55 miles without attaining homogeneity.

Besides being representative, samples should be large enough for all determinations desired. In this respect, it is better to err by collecting too much sample rather than too little. It is not unusual to want more sample for tests not originally planned, or for check analyses. When the sample must be preserved for one or more determinations, it is sometimes essential to take duplicate or triplicate samples to insure availability of complete data.

How many samples should be collected? In a symposium, "Usefulness and imitations of Samples," sponsored by the A.S.T.M., (*7*) a basis is provided for determining an adequate number of samples. Where concentration critically affects controllability or treatment costs a ***high degree of certitude*** is advisable.

There is a definite trend toward mechanical sampling and mechanical collection of samples should be combined with flow measurement.

Sample Preservation. The accuracy of the data collected is also affected by sample stability and the sensitivity of the analytical procedure employed. Waste-water samples are notoriously unstable and may be due to chemical, physical or biological characteristics. Substances that are volatile, easily oxidized, or reduced and temperature and color are susceptible to rapid changes in concentration. Solids may tend to coagulate or peptize, thus affecting the suspended solids values. In one plant concerned with removing fats and fatlike substances from waste waters the responsiveness of these substances to treatment was significantly different at the sampling point than 15 min. later in the laboratory. This experience, however, represents unusual instability.

The best antidote for such sample change is, of course, prompt analysis. Since this is seldom practical, other means are employed. The most common way to minimize changes in sample composition is refrigeration of the sample at 4° C. to 10° C. Figure 1 summarizes in a qualitative manner recommended methods of fixing waterborne industrial wastes.

Preliminary Maneuvers

Formulation of Policy. When the data assembled by the fact-finding procedures are organized, evaluated and tabulated a map of the water-problem

	Values To Be Fixed															
Preservatives	Biochemical Oxygen Demand	Suspended Solids	Relative Stability	Oxygen Consumed	Dissolved Oxygen	Nitrogen Balance	Phenol	Total Solids	Sludge	Mud	Chlorinated Waters	Cyanide	Total Solids
Sodium thiosulfate											X [5]		
Sodium benzoate										X [4]			
Refrigeration	X [1]	X [1]	X [1]	X [1]	X [1]	X [1]			X [4]	X [4]			X [1]
Sodium azide					X [1]								
Sulfuric acid					X [1]	X [1]				X [4]			
Zinc acetate							X [1,3]	X [1]					
Copper sulfate							X [3]						
Sulfamic acid					X [2]		X [3]					X	
Add NaOH to pH 7.5-8.5													
pH to 1.5						X [1]							
Chloroform				X [1]									
Formalin									X [4]				
Time limit before fixation	6-12 Hrs.	6-12 Hrs.	6-12 Hrs.	6-12 Hrs.	1-2 Hrs.	6-12 Hrs.	4 Hrs.	1-2 Hrs.	7 Days	7 Days	1-2 Hrs.	1-2 Hrs.	6-12 Hrs.
Time limit after fixation	Not Known	Not Known	Not Known	Not Known	24 Hrs.	Not Known	Few Days	Not Known	Not Known	Not Known	12 Hrs.	Not Known	Not Known

Fig. 1. Preservatives for water-borne waste samples.

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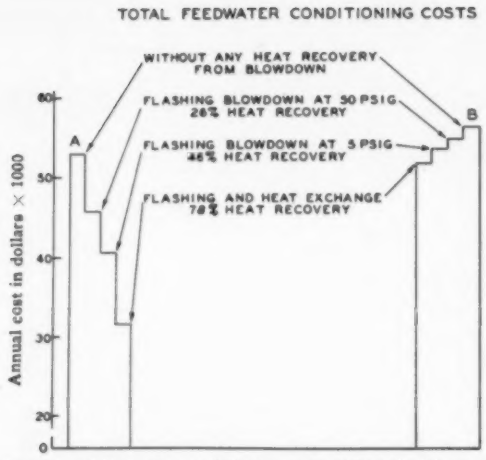

Fig. 2. Comparison of feedwater conditioning costs with and without heat recovery from blowdown.

A. Lime plus sodium aluminate for clarification and bicarbonate hardness removal plus sodium cycle softening and internal treatment.

B. Lime plus sodium aluminate for clarification and bicarbonate hardness removal plus deionization for reduction of dissolved solids and internal treatment.

areas in the plant is now available. This map will show the location, complexity, and severity of the plant's waste-disposal problems. At this point, management should be supplied with adequate information on the following points so that basic policy on pollution abatement can be established:

1. General and specific regulations on the discharge of waste waters which apply to the plant
2. Magnitude, composition and variability of the pollution load which:
   a. is discharged by plant
   b. is allowed by the regulatory body
   c. can be assimilated by receiving body of water
3. Probable costs of treatment to reduce the pollutional load to the quantity specified by 2b and 2c
4. What other industries and municipalities in the same and similar drainage areas are doing toward pollution abatement
5. Whether competitive plants are treating their waste waters

After considering the engineering, economic, legal and public relations factors, management should define its general policy on pollution abatement. Then an individual or group should be assigned the responsibility of planning and executing a program of water management and pollution abatement in consonance with this policy. Guided and supported by a well-defined policy on pollution abatement the water management group can then effectively move to minimize and simplify the waste water problem through:

1. Conservation of heat, water and materials
2. Stagewise use and reuse of water
3. Reclamation of waste products
4. Segregation of various wastes to facilitate treatment
5. Modification of processes or operating procedures

Conservation of Heat, Water, and Materials. Logically pollution abatement should begin by reducing the loss of materials to the plant's waste waters. In refineries regular "leak patrols" have been established to prevent and detect leaks. Plant suggestion boxes will often furnish ideas on how materials losses can be decreased.

Good housekeeping practices constitute an important step toward waste elimination and materials conservation. How to organize plant personnel to carry on a campaign for this purpose has been discussed by Hart (*4*) and others.

Conservation of water, heat, and materials helps to solve waste-disposal problems and generally is profitable besides. If raw and process materials do not get into the sewers they need not be removed from waste waters. However, there is neither incentive for nor advantage in water conservation where sufficient water is available for dilution of industrial wastes and where regulations do not forbid it.

In paper and pulp mills important economies in heat and materials have been realized through saveall systems. Despite this fact many plants ignore such savings until pressure for pollution abatement makes such changes imperative. In petrochemical plants valuable hydrocarbons picked up by condensing and cooling waters are increasingly recovered in separators at the units producing the wastes. If these materials are trapped in a separator near the source it is likely they can be recycled with little or no prior purification.

Figure 2 illustrates (*1*) how fuel and water costs are sometimes interdependent. Without heat recovery from blowdown, the two water-treatment costs are about equal. However, with increasing degrees of heat reclamation from blowdown, treatment *A* becomes progressively more economical than treatment *B*.

Stagewise Use and Reuse of Water. It is common to recirculate cooling water particularly in areas of limited water supply. But in general these techniques have not been exploited to the fullest economic extent. This is especially true where waste waters must be conditioned before discharge.

The treatment of waste water for reuse is especially practical if water is scarce or effluent must be treated to a purity level approaching or exceeding quality requirements for usage. For example (*3*) at a Kansas refinery 1200 gal./min. containing 250 p.p.m. of oil is passed through a softening and clarifying unit 45 ft. in diameter. About 1200 gal./min. of effluent containing 10-18 p.p.m. of oil is reused as cooling tower make-up. Chemical costs average 4 cents/1000 gal. and include ferric sulfate, soda ash, and lime. The total installed cost of the equipment was $60,000.

Sometimes minor changes in equipment or method will permit reuse of water otherwise not possible. For example, air-cooled heat exchangers are economical where make-up water is expensive to treat or scarce in supply, or where the temperature of the fluid to be cooled is relatively high. Thus, a refinery experiencing scale deposition at the hot end of a heat exchanger obtained substantial relief by stabilizing the water. Scale formation was completely eliminated by inserting a vertical loop of pipe just ahead of the heat exchanger. This loop served as an air cooler reducing the gas temperature just enough for the stabilization treatment to work perfectly.

These experiences show how water of nonideal quality can do a job in a reuse role even though it may not exactly fit the part. Sometimes the question of whether a water can be used or not can be answered only through trial. At one refinery small heat exchangers which are easy to take apart and inspect are installed ahead of the larger units. These test units bridge a gap between the open condenser box and complete reliance on laboratory control.

What are the limits of cooling water quality which can be tolerated in typical industrial cooling circuits? Inorganic and organic sulfides should be less than 1 p.p.m. Bicarbonate (HCO_3) concentrations up to 500 p.p.m. and sulfate concentrations up to 5,000 p.p.m. can be stabilized. Of course, organic growths should not be permitted in cooling circuits. Hydrogen ion concentrations, expressed as pH, may vary from five to nine depending upon treatment applied. These water quality limits are only suggested to indicate the latitude of concentrations within which recirculation can be practiced.

Reclamation of Waste Products. If the leakage of materials from process cannot be prevented every effort should be made to reclaim them. For example, one chemical plant reports that $1000 worth of fuel is recovered daily from petroleum cracking units producing ethylene. Large volumes of cooling water containing light oils, heavy oils, and tars are discharged. The heavy oils and tarlike substances are separated from the light oils in a clarifier. After purification by centrifugal separation the light oils are used as fuel in the powerhouse saving $1000/day in fuel costs. Similarly brine from the phenol plant is used as a raw material in the chlorine plant.

Use of softener sludge to assist in clarification of separator effluent has been reported by Kalinske (*6*). Waste aluminum chloride was used by Weston (*8*) for coagulation of waste water. Recently, a large plant was built by Maneely Chemical Co. to produce zinc chloride and zinc sulfate from pickle liquor and zinc dross.

It is a common refinery practice to skim oil from separators and pump it to a storage tank where three or more phases develop. The lower phase is water containing substantial oil, the intermediate phase is a water and oil emulsion and the top layer is oil. In practically all instances this top layer is suitable for direct rerun. Generally, it contains less than 1 per cent of bottom sediment and water. It is the middle layer which presents difficulties from a reuse or disposal standpoint. This so-called slop oil emulsion is solids stabilized except where petroleum soaps are present. Resolution of this slop oil was a vexing problem until the Oliver United Filter Co. succeeded in breaking these slop oil emulsions on their precoat filter.

Depending on the gravity of the oil, the water content (which may vary from a few per cent to 95 per cent) and more importantly on the amount and character of the solids present, flow rates of 1½ gal./(sq.ft.)(hr.) to 20 gal./(sq.ft.)(hr.) are obtained. After passing through the precoat filter, the mixture of oil and water is allowed to settle for about six hours. The separation is sharp and complete as shown by Figure 3. The oil is withdrawn for rerun and the water separated from the oil is returned to the waste water clarification unit. The solids removed from the slop oil emulsion are enmeshed in the precoat material and present no disposal problem. At one refinery the reclaimed oil will pay for the precoat filter installation within 30 months.

At several refineries spent caustic is used as a demulsifying agent for treating slop oil. In one refinery the spent caustic has replaced an expensive commercial demulsifying agent in the emulsion-treating operation. The recovery of salt cake in rayon plants and strong caustic in cotton mills are other examples of reclamation of waste materials.

Segregation of Various Wastes to Facilitate Treatment. Due to the many substances and widely fluctuating concentrations found in chemical plant waste waters, treatment of mixed wastes is difficult if not impossible. Consequently, segregation of wastes according to type is sometimes essential and usually advantageous. Thus, in a plating waste-disposal system, it is best to separate the chrome-bearing waste and cyanide wastes. The hexavalent chrome must be reduced and the cyanide is ordinarily oxidized. Thus, to treat the same stream for removal of both cyanide and hexavalent chromium would require a complete reversal of reaction medium from a reducing to an oxidizing environment.

Usually it is advantageous to separate the strong wastes from the weak wastes. The cost of separate sewers for segregation is offset by lower capital cost for the treatment units. Only rarely should sewer systems for industrial waste water and storm runoff be combined. Not so evident but occasionally just as important are the advantages of segregating uncontaminated water so that the former can be released directly to the stream or reconditioned for reuse.

A plant manufacturing ceramic and metallic parts was planning to install large settling tanks and auxiliary equipment for treating approximately 45,000 gal. of waste water/day. However, investigation of the plant's water-use practices, revealed that the plant wasted more than half the water it purchased. Furthermore, approximately one third of the remaining water was easily segregated and discharged without treatment. The residual waste volume requiring treatment amounted to only 6,000 gal./day. To condition this small volume for discharge, a 700-gal. settling tank was provided and a discarded plate-and-frame filter press was used to dewater the settled solids. Thus, the disposal problem in this plant was simplified, minimized, and corrected with the expenditure of much less money than would have been necessary for facilities to treat 45,000 gal. In addition the plant water bill was reduced by 50 per cent.

When strong wastes are treated, the treatment units can be smaller and treatment is usually less costly than when the same amount of a pollutant is removed from a larger volume of water.

Modification of Production Procedures or Processes. Sometimes seaboard plants can dispose of small quantities of exceptionally foul wastes by hauling the wastes by barge or sending them as ballast on outgoing vessels for dumping at sea. Occasionally it is possible to eliminate entirely or ameliorate a waste-disposal problem through changing the waste-disposal procedure. At least one refinery solved the problem of waste-caustic disposal by selling it to a pulp mill.

The most satisfactory solution, thus far suggested for the difficult problem of waste sulfite liquor disposal, is a change from calcium to magnesium or ammonium salts with almost complete recovery of the chemicals and heat. The substitution of closed coils in reaction vessels for open coils will sometimes eliminate difficult waste-water problems. A chemical plant in Maryland faced with the necessity of treating fluosilicic acid altered its processing to produce silica gel from the waste fluosilicic acid.

Frontal Assault

Development of Treatment Methods.

Fig. 3. Samples of slop oil before and after filtration through a precoat drum vacuum filter.

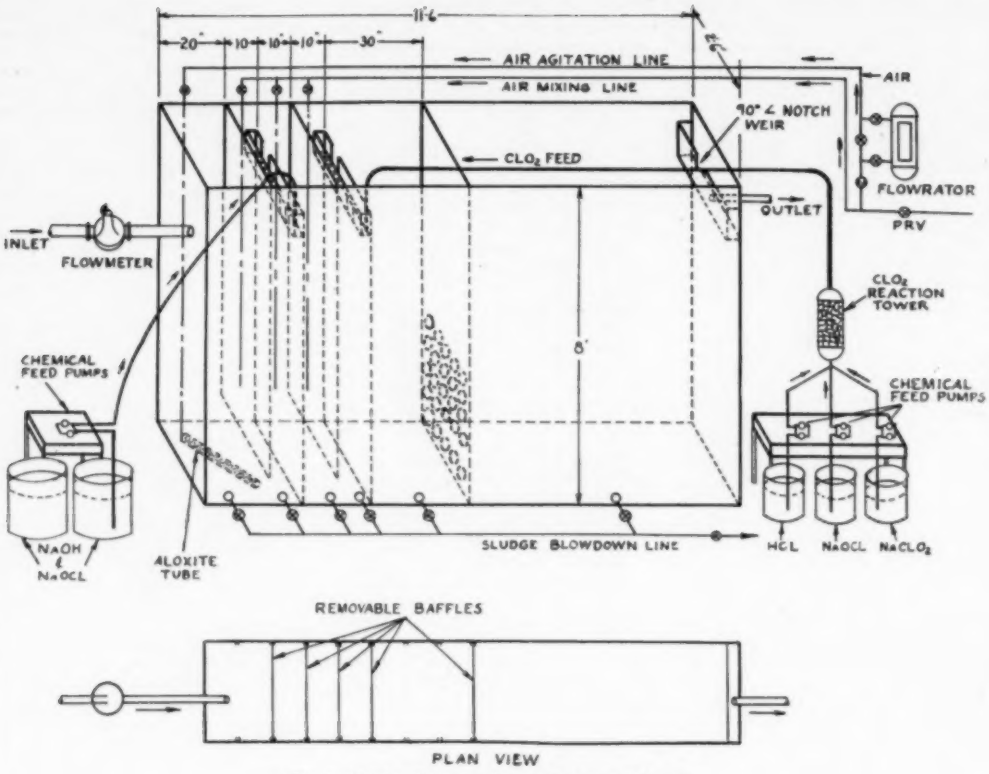

Fig. 4. Pilot unit for treatment of gas-washer water.

Only after every practical step has been taken to dwarf the plant's waste-water problem should treatment procedures for the waste water be decided upon. However, consideration must be given to possible treatment methods while other steps in the pollution abatement program are developing, otherwise simplification and minimization measures cannot be properly evaluated.

The volume and composition characteristics of the plant's waste waters are likely to be greatly reduced by the prior phases of the program. After the physical, chemical, and biological characteristics of the residual wastes are known, a thorough and complete review of prior art should be made. The unit operations of water purification and sewage treatment often provide adequate solutions to industrial waste problems, but even this reservoir of engineering experience frequently fails. Consequently, there is a great need for more investigational work to develop new water-treatment techniques. For example, hydrocarbon oxidizing bacteria are credited (*9*) with correcting oil pollution of waterways and soil. Gradually, how to employ such microbes in treating oily wastes will be known just as engineers have learned to use biological processes to treat such toxic substances as phenols, formaldehydes, and sulfides.

Some promising treatment methods will be eliminated because of space requirement. Others will be dropped because of treatment shortcomings. The remaining methods should be evaluated then on the basis of capital investment required, operating costs, and fixed charges. Frequently, it is difficult to predict treatment cost or treatment effectiveness. In such cases, laboratory experimentation and/or pilot plant studies will make possible reasonably accurate estimates. Such investigations prevent costly errors in design when the problem to be solved is nonstandard as most waste-disposal problems are.

For example, a plant manufacturing producer gas was required to treat the washer water for solids reduction and destruction of phenol and cyanide. A preliminary evaluation of possible treatment procedures revealed that two solutions for the problem appeared promising. First, the washer water could be used in a once-through system treated chemically and discharged. Second, the washer water could be recirculated in a closed system. Aeration, pH adjustment, chlorination, and finally treatment with chlorine dioxide were necessary to destroy the phenols and cyanide. Due to the high cost of chlorine dioxide and uncertainty regarding the quantity required, it was decided to determine chemical treatment costs for a once-through system by pilot plant operation.

Design features of the pilot plant are shown in Figure 4. The pilot plant was operated at 5 to 20 gal./min. for several days to stabilize chemical feeding and to eliminate the inevitable mechanical difficulties which arise. Then several weeks of steady operation followed providing sufficient data definitely to conclude that treatment of the washer water was too expensive in this case. To treat 1.25 m.g.d. involved a daily chemical cost of $47 and a daily total cost of $93. Instead a recirculating system was decided upon with clarification and cooling between washing cycles. The estimated daily cost of procedure was $28.

The pilot plant was again operated to study more closely the settling characteristics of the suspended solids, the physical characteristics of the sludge solids, etc. The pilot plant not only furnished the data for selecting the basic disposal plan but also design data for clarifying the recirculated washer water. The value of this pilot plant study is eminently evident.

The work of Weston (*8*) using duplicate coagulation pilot plant units for clarifying refinery wastes, was a valuable contribution to water technology and further demonstrates the usefulness of pilot plant studies. The treatment of formaldehyde wastes on high rate recirculating filters was studied by Dickerson (*2*) with a pilot filter. These are examples of how the frontiers of knowledge regarding the adaptability of classical sewage treatment units can be safely extended in new directions through pilot plant investigations.

Consolidation of Gains

The success of any industrial water-management program, of which pollution abatement is a part depends upon good follow-up and follow-through. Furthermore, when plant expansions or new facilities are planned, full consideration should be given to the water aspects of such plant alterations or extensions and how they affect over-all water-management program. Since the breadth and depth of water technology are increasing continually, a dynamic and sustained interest in the means and methods of pollution avoidance and abatement must be maintained in the plant. Every plant employee down to the labor level should be aware of the plant's water problems. This requires education through posters, pep talks, area prizes for good housekeeping, etc.

Summary

Industrial plants are inclined too often to refuse to meet the issue of pollution abatement until action is forced upon them by regulatory agencies. In such instances there is usually insufficient time for thorough study of the problem and a more expensive solution is reached than could have been developed over a period of years through a well-planned program of industrial water management.

Localized water shortages and generally increasing direct and indirect water costs have made piecemeal solution of industrial water problems inadvisable. Each water problem must be considered as only a part of the whole plant-wide water problem. This over-all approach to water problems is industrial water management. In many plants, industrial waste disposal is currently a major problem in water management.

Acknowledgment

Appreciation is expressed to R. E. Hall, E. P. Partridge, and C. E. Kaufman of Hall Laboratories, Inc., for assisting with this paper.

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Discussion

A. D. Potter (Texas State Health Department, Austin, Tex.): How is industrial waste management likely to affect the cost or quality of the product?

C. F. Hauck: Here is an example of how the practice of industrial water management resulted in an improvement of product. Because poor water quality was suspected as the cause of unsatisfactory sizing, a water consultant was retained to investigate water-treatment procedures and facilities in a large paper-board mill. Several methods of improving treated water quality were prescribed. At the same time the water survey uncovered the major cause of the erratic sizing. Procedures were then initiated which resulted in improving and maintaining sizing quality. It is evident that industrial plants should be alert to the *whole* water problem and not to just a part of it.

(Presented at Houston (Tex.) Meeting.)

LETTER TO THE EDITOR

"MODIFIED REYNOLDS NUMBERS"

Sir:

There is a growing practice in chemical engineering to refer to "modified Reynolds numbers." I would like to express an opinion on this subject for I think the practice is wrong.

The Reynolds number is a dimensionless ratio found to be useful in many phases of fluid mechanics. In so far as certain heat transfer operations are the results of fluid flow mechanism or are analogous to them, the Reynolds number is also a useful parameter. The Reynolds number can be derived by one of many techniques, but regardless of the manner of derivation the significant basic elements of the Reynolds number are a length characteristic, a velocity, and a kinematic viscosity. One of the most common formulations of the Reynolds numbers is Lu/ν, or $Lu\rho/\mu$. Notations for these symbols are L for length, u for velocity, ρ for density, μ for viscosity, and ν for kinematic viscosity.

Reynolds number also is interpreted to mean that it is a ratio of inertia force to viscous force. Reynolds number can also be thought of as a ratio of eddy viscosity to kinematic viscosity. However, no matter what interpretation is put upon it or what actual length or velocity may be described, it still is true that the result of the ratio is a Reynolds number. It seems to me that if someone wishes to modify the Reynolds number he would get something other than a Reynolds number. Specifically, there would be something other than a length, a velocity, and a kinematic viscosity.

Unfortunately, chemical engineers have for some reason gotten into the habit of assuming that a diameter of a pipe is the length specification for a Reynolds number. Such is far from the case. The length in the Reynolds number might be the diameter of a pipe, the length of a pipe, difference between the diameter of two concentric pipes, the distance between planes, the equivalent radius of a leading edge of an air foil, the diameter of a spherical particle, or literally a score of other length or equivalent length dimensions. Furthermore, the velocity in a Reynolds number might be an instantaneous velocity or an average velocity. In view of this it seems to me to be completely out of order to speak of "modified Reynolds number." Length, velocity, and kinematic viscosity in proper ratio constitute a Reynolds number no matter what length is described, what velocity is described, or what kinematic viscosity is described.

Let us therefore have done with the extraneous and misleading prefix "modified."

J. H. Rushton,
Chicago, Ill.

June 4, 1951.

CAST ALLOY REFERENCE SHEET

N. S. MOTT, Chief Chemist Metallurgist
The Cooper Alloy Foundry Co., Hillside, N. J.

ALLOY: ABRASION, HEAT AND CORROSION RESISTANT HARDENABLE 12% CHROMIUM ALLOY.

APPLICATIONS AND REMARKS: This composition is a mildly corrosion resistant alloy capable of being heat-treated for a variety of properties including high strength and hardness. It has a low coefficient of expansion, is readily machineable and weldable, and finds extensive use in valve bodies, valve trim, pump parts, grinding units, etc., especially in power plants, oil refineries, and paper mills. The alloy has suitable corrosion resistance for applications involving superheated steam, hot oil, foodstuffs, and some neutral salts; however, it will rust to a slight extent in a moist atmosphere. Its low cost, high strength, high hardness, and wear resistance, coupled with an appreciable amount of corrosion resistance, make it an ideal alloy for specific applications where these factors are of primary importance.

CHEMICAL COMPOSITION RANGE: C < 15%; Cr 11.5-14%; Ni < 1%; Si < 1.5%.

DESIGNATIONS: AC1 CA-15; A1S1 410; SAE 60410; ASTM A296-49T, Grade CA-15.

MACHINABILITY: This alloy is readily machinable, and to accomplish this, it should be in the hardness range of 220-240 BHN which is best obtained by tempering at 1200° F. Softening to too low a hardness causes gumming and poor machinability.

HEAT TREATMENT: Heat treatment for this alloy consists of oil-quenching or air-cooling from 1800° F. after slowly heating to temperature and holding for 1 hr., followed by tempering for 3 to 4 hrs. at the correct temperature necessary to obtain desired properties. Tempering at 600° F. gives best strength and corrosion properties.

WELDABILITY: When welding with a rod of similar composition preheat casting to 400° F. After welding cool to not less than 300° F. then heat to 1350° F. for 4 hrs. and air cool. If casting cannot be heated use a 25-20 type welding rod. As the alloy is air-hardening there is a great tendency towards cracking during welding and considerable care must be taken.

MECHANICAL AND PHYSICAL PROPERTIES:

Tensile Strength, 1000 lb./sq.in.	197*
Yield Point, 1000 lb./sq.in.	139*
Elongation, %	16*
Reduction in Area, %	42*
Brinell Hardness	372*
Charpy Impact (Std. Keyhole ft.-lbs.)	13*
Med. of Elasticity (× 10^6 lb./sq.in.)	29
Specific Gravity	7.67
Melting Point	2700-2790° F.
Specific Heat (B.t.u./(lb.)(° F.)) 32-212° F.	0.11
Thermal Expansion (× 10^6 in./(in.)(° F.)) 32-212° F.	5.5
Thermal Conductivity (B.t.u./(hr.)(sq.ft.)(° F./in.)) 32-212° F.	174
Electrical Resistance (Ohms./cir. mil. ft.) @ 32° F.	364

* Hardened and stress relieved @ 600° F.

CORROSION RESISTANCE

ACIDS	
Acetic 5%, 70 F	G
Acetic 5%, boiling	F
Acetic 80%, 70 F	N
Acetic 80%, boiling	N
Acetic Glacial, 70 F	N
Acetic Glacial, boiling	N
Benzoic 5%, 70 F	E
Boric 5%, 176 F	E
Chromic 10%, 70 F	N
Chromic 10%, boiling	N
Chromic 50%, boiling	N
Citric 5%, 70 F	E
Citric 25%, boiling	N
Citric 50%, boiling	N
Formic 5%, 70 F	N
Hydrochloric 1%, 70 F	N
Hydrochloric 1%, boiling	N
Hydrochloric, 5%, 70 F	N
Hydrochloric 5%, boiling	N
Hydrochloric 25%, 70 F	N
Hydrochloric 25%, 176 F	N
Hydrofluoric 48%, 70 F	N
Hydrofluoric 48%, 176 F.	N
Lactic 5%, 70 F	P
Malic, all tempe.	P
Nitric all conce., 70 F	G
Nitric 65%, boiling	P
Oleic all conce., all tempe.	F
Oxalic 5%, boiling	P
Phosphoric 10%, 70 F	N
Phosphoric 85%, 70 F	N
Phosphoric 80%, boiling	N
Stearic concentrated to 200 F	G
Sulfuric 2%, 70 F	N
Sulfuric 2%, 176 F	N
Sulfuric 2% boiling	N
Sulfuric 5%, 70 F	N
Sulfuric 5%, 176 F	N
Sulfuric 5%, boiling	N
Sulfuric 10%, 70 F	N
Sulfuric 10%, 176 F †	N
Sulfuric 10%, boiling	N
Sulfuric 78% (60° Be), 176 F	N
Sulfuric 93% (66° Be), 70 F	G
Sulfuric 93%, 300 F	N
Oleum, 70 F	P
Mixed Acids 57% H_2SO_4, 28% HNO_3, 176 F	P
ALKALIES	
Ammonium Hydroxide, all concs.	E
Calcium Hydroxide 10%, boiling	E
Calcium Hydroxide 50%, boiling	P
Sodium or Potassium, Hydroxide, all concs., 70 F	E
Sodium or Potassium Hydroxide < 20%, boiling	G
Sodium or Potassium Hydroxide 30% boiling	P
Potassium, Molten, 600 F	N
NEUTRAL AND ALKALINE SALTS	
Barium Sulfide, 70 F	G
Calcium Chloride 5%, 70 F	P
Calcium Sulfate Sat., 70 F	E
Magnesium Chloride 5%, 70 F	N
Magnesium Sulfate 5%, 70 F	E
Sodium Carbonate, all concs., 70 F	E
Sodium Chloride 5%, 70 F	F †
Sodium Sulfate 5%, 70 F	G
Sodium Sulfide 5%, 70 F	F
Sodium Sulfite 5%, 70 F	P
ACID SALTS	
Alum 10%, boiling	P
Aluminum Sulfate 10%, 70 F	N
Ammonium Chloride 5%, 70 F	F †
Ammonium Sulfate 10%, 70 F	P
Ammonium Sulfate 10%, boiling	N
Ammonium Nitrate, all concs., 70 F	G
Stannous Chloride 5%, 70 F	N
Zinc Chloride 5%, boiling	N
OXIDIZING ALKALINE SALTS	
Calcium Hypochlorite 2%, 70 F	N
Sodium Hypochlorite 5%, 70 F	N
Sodium Peroxide	G
OXIDIZING ACID SALTS	
Ammonium Persulfate 5%, 70 F	F
Cupric Chloride 1%, 70 F	N
Cupric Sulfate 10%, 70 F	E
Ferric Chloride 10%, 70 F	N
Ferric Sulfate, boiling	F
Mercuric Chloride 2%, 70 F	N
Stannic Chloride 5%, 70 F	N
WET AND DRY GASES	
Chlorine Gas Dry, 70 F	G
Chlorine Gas Wet, 212 F	N
Sulfur Dioxide Dry, 575 F	E
Sulfur Dioxide Wet, 70 F	F
Sulfur Dioxide Solution, 70 F	P
Sulfur Dioxide Spray, 70 F	N
Hydrogen Sulfide Dry	G
Hyrdogen Sulfide Wet	F
ORGANIC MATERIALS	
Acetone, 70 F	F
Acid Sludge (50% H_2SO_4), 200 F	N
Alcohol—Methyl and Ethyl	E
Analine Hydrochloride, 70 F	N
Benzol, 176 F	F
Carbon Tetrachloride	E
Chloroform	F
Ethyl Acetate, 70 F	G
Formaldehyde, 70 F	E
Phenol 5%, boiling	G
Refinery Crudes	N
Trichlorethylene, boiling	E
PAPER MILL APPLICATIONS	
Kraft Liquor	G
Black Liquor	G
Green Liquor	G
White Liquor	G
Sulfite Liquor, 176 F	P
Chlorine Bleach	N
Paper Makers Alum	P
PHOTOGRAPHIC INDUSTRY	
Humid Atmospheres	P
Cellulose Acetate	P
Acetic Anhydride	N
Acetic Acid + 1% H_2SO_4	N
Developers	E
Solutions Containing SO_2	P
Silver Nitrate, 70 F	E
FERTILIZER MANUFACTURING	
H_3PO_4 + H_2SO_4 + HF	N
PICKLING OPERATIONS	
H_2SO_4 + Dichromate, 176 F	N
H_2SO_4 + Hcl, 176 F	N
CORROSIVE WATERS	
Acid Mine Water	N
Abrasive Acid Mine Water	N
Sea Water	G
Brackish Water	F
FOOD & ASSOCIATED PRODUCTS	
Brines	F
Edible Oils	G
Fats	G
Fatty Acid Distillation	F
Fruit Juices	G
Ketchup	F †
Milk Pasteurizing	F
Vinegar and Salt, 70 F	P

RATINGS:

E—Excellent resistance. 0.001 max. in. per year of penetration. Corrosion so slight as to be harmless.
G—Good resistance. 0.001-0.010 in. per year of penetration. Satisfactory service expected; at most a slight etch.
F—Fair resistance. 0.010-0.120 in. of penetration per year. Satisfactory service under specific conditions. Light to moderate attack.
P—Poor resistance. 0.120-0.380 in. of penetration per year. Satisfactory for temporary service only.
N—No resistance. 0.380 min. in. of penetration per year. Rate of attack too great for any use.
†—Subject to pitting type corrosion.

No. 1
(FIRST OF A SERIES OF TEN)

PHOTOGRAPHIC EXHIBIT

A Feature of the Rochester Meeting

SEPTEMBER 16-19

THE next Institute meeting is to be held Sept. 16-19, 1951, at Rochester, N. Y., "the Photographic Capital of the World." A symposium on "Industrial Applications of Photography" is scheduled for one of the technical sessions and it is planned to supplement this with an exhibit showing applications of photography in chemical process industries in general. (*See* August issue for complete details of Rochester meeting.) Photography is being used increasingly in industry to show operations, to teach, to simplify, to explain, and to solve. It is some of these applications and functions in industry that the Rochester exhibit aims to show. Black and white, and color, still, and motion pictures will be shown.

Photography has been of service to many A.I.Ch.E. members, both from a functional and a report standpoint. Functional photography has helped to show how things work and operate and to record a movement often invisible to the eye. From a report standpoint, photography has been used increasingly to describe and emphasize various aspects of chemical engineering reports. It has and will continue to eliminate the necessity for certain art work, diagrams and flow sheets. The Rochester meeting photographic exhibit aims to show these two uses to its members: functional and report photography.

If you have a particularly interesting example of photography in industry of a functional or report type, we would like to display it at Rochester.

The following suggestions are made regarding prints:

1. Prints should be mounted on a board, 16 in. × 20 in. size.
2. Prints must be large enough to be seen from a reasonable distance.
3. They must be properly identified as to what is shown or what is happening. In many instances, a picture is not worth 1000 words unless properly identified and titled in a legible manner.
4. Your address should be included on the back of all submitted prints.
5. Send to: Hotel Sheraton, 111 East Ave., Rochester, N. Y., Attention: J. S. Bruce, Hold for A.I.Ch.E. meeting.

It is also hoped that high speed and normal motion pictures will become a continuous part of this exhibit. Again, you may have, or know of something that would be of general interest. Here, the following points should be considered:

1. It must be titled.
2. It should be brief.
3. It must be 16-mm. motion picture film, either silent or sound.
4. Your address should be included.
5. Forward as mentioned above.

All prints must be received before Sept. 1, 1951. These prints will be returned to the owners at the close of the meeting.

Your help in forwarding interesting photographic results from the field, plus material now in progress will assist in preparing a meeting "stopper." While no prizes are to be awarded, possibly the acceptance of your print or film in the Chemical Engineering Technical Salon will prove interesting and useful to other A.I.Ch.E. members.

Photograph above shows downtown Rochester.

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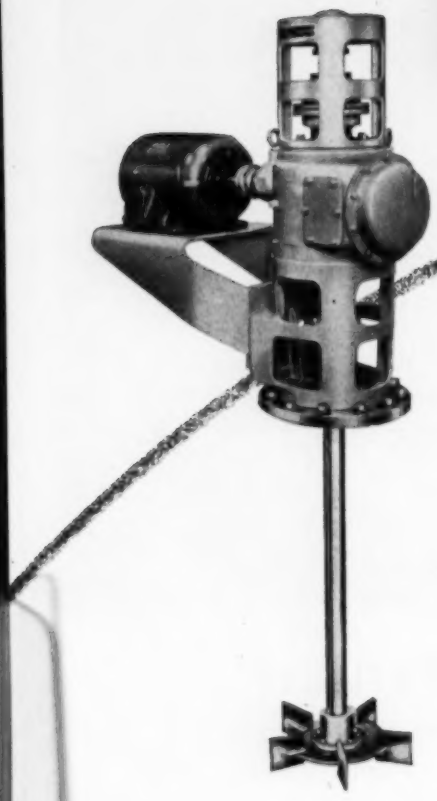

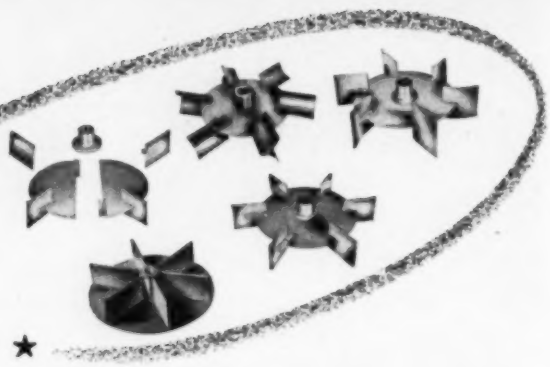

LIGHTNIN SERIES "E" heavy-duty turbine agitator. Made in sizes from 1 to 500 HP. Quickly convertible for open-tank or closed-tank operation. Operates at any one of 16 standard AGMA speeds. Speed is quickly changed without removing unit from tank. Described in Catalog B-36.

For Fluid Agitation

that does what you want it to do

Flexibility—the kind that saves you money and helps you get good processing results—is built into Lightnin Mixers.

One example of this is the Lightnin impeller. Sizes and shapes are standard—with *known* flow pattern and power response.

As blades are added, the power requirement increases, stepwise, in a *known* quantity.

This means that Mixco engineers can *accurately predict* what your process results will be. It also permits accurate *sizing* of the mixer—big enough to do the job without overloading, small enough to keep your horsepower requirements at a minimum.

The impellers on Lightnin Mixers are made of standard, interchangeable parts. Blades can be added, changed, replaced without going to the expense of a new impeller. This cuts upkeep and gives you *flexibility* to meet future changes in process requirements.

To get the whole story of how Mixco engineers and Lightnin Mixers can give you fluid agitation that's *guaranteed* to do what you want it to do, write today.

MIXING EQUIPMENT Co., Inc.

(MIXCO)

199 Mt. Read Blvd., Rochester 11, N. Y.

In Canada: William & J. G. Greey, Ltd., Toronto

Please send me the literature checked:

- ☐ B-76 Side Entering Mixers
- ☐ B-78 Top Entering Mixers (Propeller Type)
- ☐ B-89 Top Entering Mixers (Turbine and Paddle Type)
- ☐ DH-50 Laboratory Mixers
- ☐ B-36 Condensed Catalog showing complete line
- ☐ B-75 Portable Mixers (Electric and Air Driven)

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LIGHTNIN PORTABLE MIXERS

World's largest-selling line. Electric or air motor, direct or gear drive. Sizes ⅛ to 3 HP.

LIGHTNIN SIDE ENTERING MIXERS

For tanks up to 5 million gallons. Gear motor or V-belt drive; motorless types. Sizes 1 to 25 HP.

LIGHTNIN TOP ENTERING AGITATORS

Turbine, propeller and paddle types. For open or closed tanks. 16 speeds available in new heavy duty drive. Sizes ¼ to 500 HP.

A COMPLETE LINE . . . UNEXCELLED TECHNOLOGY

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BULLETINS

1 • DISINTEGRATORS. Disintegrators for the process industries are illustrated in a folder by the Rietz Manufacturing Co. Construction details are shown in a cutaway view of the model, a fixed-hammer type. Series ranges from 5 to 400 hp.—five sizes, two speeds and 34 models. For particle size reduction, crushing, grinding, pulverizing, shredding, etc.

2 • VERTICAL SUMP PUMPS. Four new vertical sump pumps are described in a recent bulletin of The Deming Co. Range of sizes from 1 in. to 10 in. discharge, capacities from 10 to 3000 gals./min., and heads up to 140 ft. Electric motor or steam turbine drive. Bulletin gives details on sizes, construction, installations, rating charts, etc.

3 • VALVE CATALOG. A detailed description of the operation and performance of The Annin Co. valves, including the Domotor, electro-pneumatic, handwheel, and combination valves. Excellent illustrations and text show valve designs, operating characteristics, installation ideas, flow flexibility, packing features, and assemblies for complex problems in the processing industries. Available in alloy constructions. Domotor valve is pressure-operated, acts on a supply pressure of 25 to 50 lbs./sq. in.

4 • METERS. Rockwell Manufacturing Co. with a new bulletin on the selection of meters for measuring chemicals, petroleum products and other liquids, with varying corrosive characteristics. Range in size from ½ to 3 in. Bulletin gives operation characteristics, general specifications, counters reach seven figures. Cases are cast iron, bronze, and steel; chambers of bronze and ni-rest; pistons are bronze, ni-rest, aluminum, carbon, and rubber. Bulletin gives dimensions, details of flanges, accessories, strainers, remote registration unit. Table gives recommended materials of construction for various corrosive chemicals.

5 • ROD MILLS. For fine crushing and grinding, Hardinge Co., Inc. new catalog on rod mills. Illustrated, gives correct applications, general design, explanation of grinding action for both wet and dry grinding, construction, as well as table of specifications for mills which range from 2-ft. diameter to 11½-ft. diameter. Bulletin covers conical, flanged-end, and convex-head types, both trunnion overflow and peripheral discharge.

6 • COOLING TOWERS. Natural draft cooling towers, their construction, features, applications, etc., as practiced by The Fluor Corp., Ltd. Gives details of bowed-deck design, aerator panel design and gusset-type construction. Final section of the bulletin contains technical detail on method of estimating tower size and description of three technical articles available from the company.

7 • SCHEDULE 5 STAINLESS. A set of data sheets from The Carpenter Steel Co., which explains details of stainless steel pipe having the same outside diameter as standard schedule 40, but having a larger inside diameter. Chart shows the difference in diameters as well as a comparison of cost per foot for standard new schedule 5. Pressure test results, and data table comparing the new schedules with the older for pressures, internal cross-section, weight pounds per lineal ft., etc.

8 • DELAYED COKING. Lummus Co. has published a new brochure covering the delayed coking process. Describes the Pan-Am Southern Co.'s El Dorado unit. Economics of coking, flow chart, specifications of petroleum coke, operation of the unit (which has largest coke drums ever installed). Well illustrated. Shows method of removing coke, etc.

9 • LABORATORY BLENDER. A 4-page catalog bulletin describing the four- and eight-quart twin-shell laboratory blenders made by The Patterson-Kelley Co., Inc. Principle same as p-k production models. Blender V-shaped with materials added through the ends of each arm. Transparent plastic shells, resistant to corrosion.

10 • ANALOG COMPUTERS. A catalog and manual on high speed, all electronic analog computers for research and design, from G. A. Philbrick Researches, Inc. Origin and development of electronic analog computers applicability, mathematics, instruction, are all explained, as well as components that make the computation. For the engineering

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group that has difficult mathematical problems. Explains the adding, integrating, differentiating, squaring, square root, etc., components. Assemblies, constructions, plus operating and philosophical instructions all included in this complete discussion of analog computers for research and design.

11 • *VISCOSITY PACKAGE.* A packet complete and up to date with data on viscometers made by Brookfield Engineering Labs., plus information on the general subject of viscosity. Contains catalog, data sheet, price lists, plus a list of available reprints.

12 • *CONTROLLED GRANULATION.* For granulating chemical products, a bulletin of Allis-Chalmers. Includes various types of roller mills for single or multiple-stage grinding, sifters for grading through coarse, medium, or fine meshes, flaking mills, double-drum dryers. Gives typical mill flow charts showing how equipment is lined up for specific job.

14 • *LIQUID-PROCESSING EQUIPMENT.* Catalog describes entire line of stainless steel filters, mixers, tanks, and pumps of Alsop Engineering Corp. Sealed-Disc filters are illustrated with a sample of asbestos filter discs. Equipment is illustrated. Performances, dimensions, constructions, capacities, etc.

15 • *DENSITROL.* For continuous measurement and control of liquid density, a Precision Thermometer & Instrument Co. bulletin on Densitrol. Instrument uses a chain-weighted plummet as indicating element. Position can be determined by an electrical position-measuring system which, in turn, can indicate, record, and control liquid density. Bulletin gives performance characteristics, shows the standard pilot plant model, pipe-line model, and direct indicating model. Gives materials of construction, pressures, temperatures, etc., of use.

16 • *PLASTIC PREFORMER.* Advantages of preforming plastics and conditions under which preforming is desirable in a brochure of F. J. Stokes Machine Co. Bulletin is technical, covers the properties of preforms, methods of preforming, punches and dies, description of preforming presses. Shows relationship between screen analysis of material and pill weight, between pressure and density of general purpose phenolic material, and between preform hardness of material and temperature.

17 • *REFRACTORIES AND INSULATIONS.* For the engineer with a problem in heat insulation or refractory cements, catalog from Refractory & Insulation Corp. should be of help. Covers refractory cements, how used, properties, casting techniques for services at high temperatures. Data on physical properties, working qualities, shipping weights. Second section covers plastic fire brick, insulating blocks, general wool insulation, etc. Prices, considerable data on the use such as steam plants, coke-oven plants, dryers, furnaces, etc.

18 • *RECIPROCATING PUMP.* A bulletin from The Aldrich Pump Co. on a 3-in. stroke triplex direct flow pump. Works at 50 hp. and pressures up to 3000 lbs./sq.in. Application to all fields handling lean oil, butane, propane, crude and gathering lines, salt water disposal, and in general industry for die-casting, plastic-molding, synthetic processing involving high pressures, etc. Hydraulic medium can be water, or hydraulic oil. Can be fitted to operate automatically with oil accumulators.

19 • *AIR CONTROL VALVE.* An illustrated bulletin, describing valves available from Ross Operating Valve Co. Hundreds of standard models including straightway, three-way, four-way, etc. Units in various sizes for hand, foot, solenoid, pilot, remote control, cam, speed control, shut-off, quick exhaust, etc.

20 • *VERTICAL TURBO DRYER.* Units for drying, sublimation, oxidation, and cooling of fragile or heat-sensitive materials, in brochure of Wyssmont Co. Vertical type, has continuous transfer of material, fans circulate air over a series of rotating, horizontal annular shelves. Discussion of materials, temperatures, applications and construction.

EQUIPMENT

25 • *VARIABLE DRIVE.* A new, larger, Speed-Trol electric power drive has been added by Sterling Electric Motors, Inc., in its standard line. The unit has positive adjustment of pulleys, infinite speed variation, and accurate control of speed under

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varying loads. Available in splash-proof and totally enclosed fan-cooled models. Can be supplied with manual control, electric, or mechanical remote control.

26 • *UNIT DUST CONTROLLER.* For controlling plant dust at isolated spots or at points far from central dust control systems, The Day Co.'s new unit dust controller. Incorporates Dual-Clone dust separator and exhauster. Operates at 6-in. external static pressure. Available in four sizes, from 500 to 2000 cu.ft./min., with or without secondary filters.

27 • *AIR FILTER.* Cambridge Corp. is now producing in quantity, an air filter originally developed for the Atomic Energy Commission and capable of removing better than 99.98% of all dust, smoke, fumes, radioactive particles, and spores. Filter is in two sizes with rated capacities of 500 and 850 cu. ft. of air/min. Dimensions of the filters are 24 x 24 x 5⅞ in. deep for the 500 cu.ft./min. size and 11½ in. deep for the 850-cu. ft. size. Static pressure of the filter when new is less than 1 in. of water. Individual units may be arranged in multiple banks in ventilating or air-conditioning units.

28 • *PLASCOR.* For laboratories, pilot plants, offices, chemical plants, etc., the U. S. Stoneware Co. has developed a floor tile made from Tygon plastic and resin-impregnated cork. Bulletin gives details of construction, sizes, colors, application, etc. Possesses the resistance of Tygon against corrosives, greases, alcohols, etc.

29 • *HEAVY DUTY MIXER.* For processing and blending viscous compounds such as plastics, heavy chemical solutions, L. O. Koven & Brother, Inc., are producing a new, heavy duty mixer, 36 in. in diameter, steam-jacketed. Has an 18-in. diameter turbine-type propeller which drives at approximately 125 rev./min. with a 2-hp. motor. Outlet valve at the bottom of the tank, thermowell, and baffles are built into the tank.

30 • *CALIBRATED DIAL.* For use with Leslie Co. temperature regulators, a new, calibrated dial for quick temperature settings. Dial fits in place of a standard adjusting sleeve and is easily installed on class T and class M regulators already in service.

31 • *PLATFORM BEAM SCALE.* For fast, low-cost industrial weighing applications, The Yale & Towne Mfg. Co.'s new platform beam scale. Designed primarily for heavy duty industrial applications where shock-loading is the rule. Lever system is all steel as are other key parts. Poises on main bar are mounted on roller bearings and end-loading platform gives the same reading regardless of the location of load. Pit requirements are only 11 in., built-in capacities up to 6400 lbs.

32 • *FIRE EXTINGUISHER.* A 4-lb. dry chemical extinguisher with rubber hose by the Ansul Chemical Co. Operating range 12 to 15 ft. and has a B2, C2 rating from the underwriters. Fully charged weighs 10½ lbs., hand-operated model has a self-closing nozzle to make the unit watertight.

33 • *AIRLOCK FEEDER.* For processing clay, diatomaceous earth, calcium carbonate, carbon black, salt, etc. Prater Pulverizer Co. has designed a new airlock feeder. For pneumatic-materials handling, the device will provide a positive method of feeding powdered and granular products into air-conveying lines, seal off air leakage at the discharge of dust collectors, and feed material at a predetermined rate into mixers, blenders, and hoppers. Models ⅓ and ½ hp. with a cast iron body and rotor. Sealed bearings.

34 • *DELAYED ACTION RELAY.* For use with diaphragm-control valves operating in series, Black, Sivalls & Bryson, Inc., are producing a new relay with a body and diaphragm case of cast bronze and stainless steel inner valve assembly with neoprene diaphragms. A spring of sufficient power delays output port opening until loading pressure has been reached, and only loading pressures above the permanent absorbed spring load will affect relayed ratios.

35 • *TUBULAR HEATERS.* For providing a low density electric heat for oils, tars, resins, and other viscous fluids, Martin-Quaid Co.'s new line of tubular heaters. Design permits large input capacity without exceeding critical heat limitations of the

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fluid, thus avoiding local overheating, carbonization, etc. Bulletin available which gives information on selection and heat density for applications.

36 • ELECTRIC BOILERS. Low-pressure boilers in the 1½ hp. range, with a maximum pressure range of 15 and 50 lbs./sq.in. using electric power, are newly designed units of Livingstone Engineering Co. New series boilers are 14 in. wide, 22 in. long, and 28 in. high and require 15 kw. input for maximum steam output. Heat output of 50,000 B.t.u./hr. Heat is generated by resistance of the boiler water to the flow of current between solid metal electrodes. Pressures are instantly adjustable.

37 • CATALYST VALVE CONTROL. Conoflow Corp. has made a special design of a Conorotomotor to operate catalyst slide valves by throttling control service. Pneumatically driven and used for handling spent, regenerated catalyst, back pressure control of flue gases, etc., on catalytic cracking units. The control operates through a gear drive and threaded stem. Permits positioning to within 1/500th of valve stroke.

38 • MINIATURE INDICATORS AND CONTROLS. For reducing space requirements on control panels, Bailey Meter Co. has developed a new line of miniature indicators and control units under the trade name MINI-LINE. Space savings up to 87%. Company line of multipoint indicators, selector valves, and remote manual relays.

39 • HAND PUMP. Hand pumps in 7, 10, 14, and 28-gals./min. capacity in several models are newly in production by Blackmer Pump Co. Used for pumps and skid tanks. Pumps have a drip-pen for barrel mounting, floor stand for underground tanks, brackets for wall or foot mounting, locking device. Larger models are geared hand pumps, the smaller being direct drive.

40 • KNEADER-MIXER. A quick dumping heavy duty kneader and mixer is in production by Charles Ross & Son Co. The unit is double-bladed with different stirrers available for operation at the same or different speeds. Dumping is accomplished by manual, hydraulic, or motorized tilting. Jacketed for cooling or heating. Is available in a variety of sizes up to 150 gals. For pilot plant use small, 1-pt., 1-qt., and 2-gal. models are available.

CHEMICALS

45 • G.I. PAINT SPECS. For paint manufacturers and technologists, Rohm & Haas 32-page book of 16 suggested formulations to meet U. S. Government paint specifications. Covers various enamels, primer, alkyd resin solutions, protective interior lacquers, formaldehyde baking enamels, zinc chromate primer, etc. Formulation is suggested for ball mill grind, mixes are given, plus the physical constants.

46 • TALLOW CONCENTRATION CHART. American Mineral Spirits Co. has a tallow miscella concentration chart covering mixtures of tallow and commercial heptane. Of interest to processors in connection with design calculation, plant operations and interpretation of operation data. Chart gives per cent tallow by weight for specific gravities at a range of temperatures in commercial heptane.

47 • PHOSCOTE PROCESS. Chicago Bridge & Iron Co. describes its new phoscote process of pickling and plating steel plate, angles, channels, etc. The process removes mill scale with sulfuric acid, plus wash and a phosphoric acid treatment. This provides a dry surface of iron phosphate which improves the bond between prime coat of paint and steel. Illustrated, shows process completely.

48 • COMPRESSED GAS PRICES. A price listing of compressed gases from the Ohio Chemical & Surgical Equipment Co. Covers 32 different gases, eight different cylinder sizes. Gives weight, cubic foot, and price. Tables show properties of gas.

49 • LINSEED GRINDING OIL. Archer-Daniels-Midland Co. in a new technical bulletin describes two grinding oils developed for paint manufacturers. Pure linseed fortified with polymerized linseed. Bulletin gives characteristics and specifications, shows test panels prepared with the new oils, and gives various formulas for making paints to federal specifications, etc.

50 • ANTI-RUST PIGMENT. Lead cyanamide for use in the protection of anti-rust paints, is now available through Millmaster Chemical Corp. Used for years in Europe, company is distributing the cyanamid compound made in Germany.

51 • BUTYL STEARATE. A technical service report on butyl stearate which gives the physical properties, shipping information and uses. From the Witco Chemical Co. Used in the protective coating, plastics, textile, paper, ink, lubricating, cosmetic, etc., industries.

52 • BRUSH AND WEED CONTROL. Complete data on methods and results in chemical brush and weed control as practiced by industrial and agricultural organizations. New operations manual from DuPont. Covers Ammate, 2,4,5-T, 2,4-D, and TCA.

53 • SPONGE RUBBER BLOWING AGENT. For producing sponge rubber, Ansul Chemical Co. has introduced Ansul Blo a bicarbonate-type material, nontoxic, resistant to water absorption, uniform size. Equal to twice quantity of sodium bicarbide. Data sheet gives features, suggested formula for production of sponge rubber.

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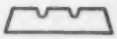
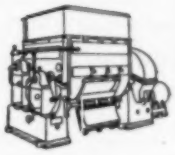
AIRPLANES NEED ALUMINUM

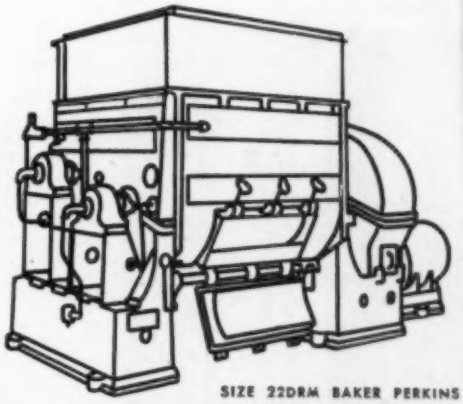


ALUMINUM PRODUCTION NEEDS CARBON ELECTRODES

ELECTRODES REQUIRE MIXING

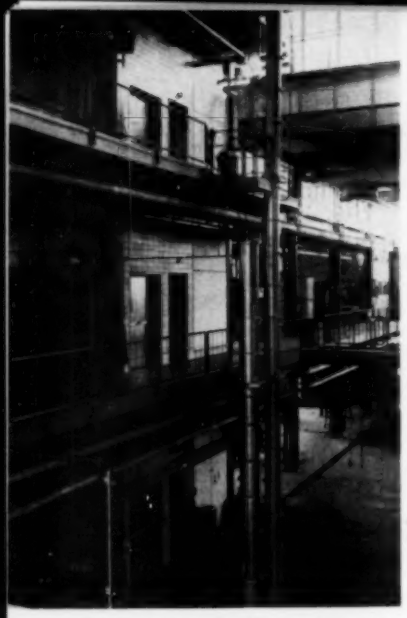


Baker Perkins unidor mixers are designed, built and preferred for the rapid processing of carbon electrodes and pot linings for aluminum production either Soderberg or Hall process. For complete confidential data write to Baker Perkins Inc. Chemical Machinery Div., Saginaw, Mich.



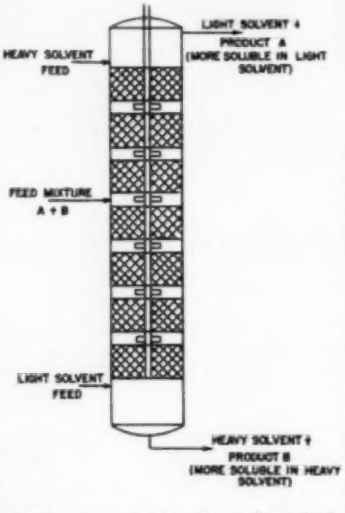
SIZE 22DRM BAKER PERKINS UNIDOR MIXER WITH CAST SIGMA BLADES CORED FOR STEAM.

SCHEIBEL EXTRACTORS IN PHARMACEUTICAL PRODUCTION

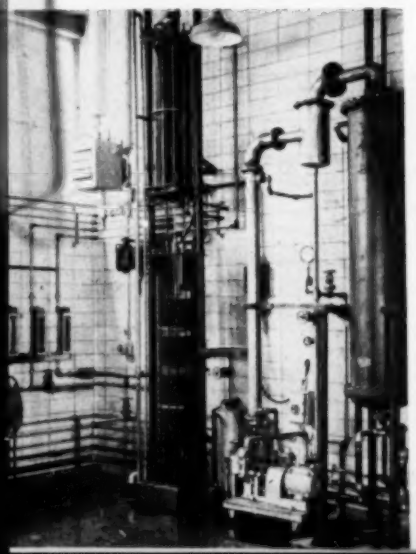


▲ Shown here are the two large pilot plant columns for fractional liquid extraction, in the Hoffmann-La Roche chemical engineering laboratory. Motor drive for the smaller 16-in. column is shown supported on a separate platform abutting from the third mezzanine.

Schematic diagram showing flow of ► feed and solvents through a Scheibel extractor.



A typical all-glass two-column pilot plant extractor unit. The upper column has three stages, and the lower five, and arrangements are made so that the unit can be either used in series or individually. ▼



LAST month at the Nutley (N.J.) plant of Hoffmann-La Roche an editorial inspection was held by officials of the company, to demonstrate the use of chemical engineering techniques and equipment in pharmaceutical manufacturing. The use of the newer chemical engineering tools in the pharmaceutical field has shown a marked increase in the past ten years, officials of the company said. One of the newer tools featured and now being used extensively in production is the Scheibel extractor, developed at the Hoffmann-La Roche chemical engineering laboratory by E. G. Scheibel. This was reported for the first time anywhere, in Chemical Engineering Progress, September and October, 1948, Volume 44, and supplies an example of how modern chemical engineering techniques are springing from theoretical development into full-scale plant use within a few years.

The tower consists of a cylindrical shell with an internal rotor shaft and knitted packing spaced at intervals. In the area between the packing, agitators mounted on the shaft, mix the solvents and feed. Agitators are belt-driven, and recoverables are removed from solvent either by subsequent distillation or processing in another extractor if the solvent carrier is not satisfactory for further production steps.

Efficiencies of the unit continue as high or higher than was reported in the original articles, and the Hoffmann-La Roche plant makes use of a good dozen full-scale production units. Materials of construction vary, depending upon the corrosive media used. In the Hoffmann-La Roche plant there are two 18-in. diameter copper units, one steel shell with stainless steel packing, 12 in. in diameter, and several polythene units using various packings such as stainless steel, silver, etc. The polythene units are self-supporting with the exception of the top agitator motor drive, which is usually supported externally by overhead hanging brackets. In one of the columns processing a mixture of corrosive acids, silver packing is used. Fittings are used with polythene gaskets, all-glass sight sections, and saran piping. The polythene columns are heat-welded by a stream of hot nitrogen, and the columns are formed from flat sheets. Pipe fixtures are installed and heat-welded into place before the column is formed. All units are four-bladed agitators, rotated from 100 to 500 rev./min., the speed being determined by solvent properties.

Production Comparisons

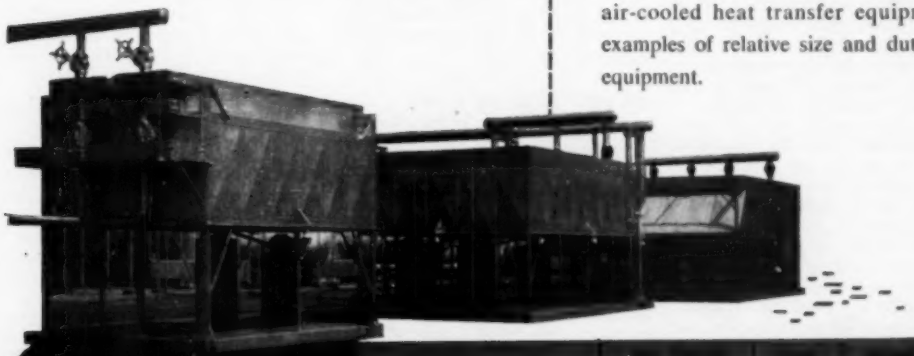
The efficiencies of the Scheibel unit are high. One 18-in. diameter copper unit now takes the place of four 1,000-gal. batch extractors, plus an evaporator unit for concentration. The particular installation is removing, by means of selective solubilities, a desired compound from an aqueous mixture. In practice, 100 gal./hr. of feed (containing 400 lb. of desired component, 300 lb. of unwanted material) is processed in the column with 400 gal./hr. of solvent. The unit has seven operation stages, and is about 12 ft. tall.

In the chemical engineering laboratory there are several extractors of various sizes and constructions, armored glass units serving well for development techniques. For pilot plant work, however, two Monel columns illustrated here have been constructed, one 42 ft. high, 14 in. in diameter, and having 30 extraction stages, the other 21 ft. high, 16 in. in diameter, and having 12 extraction stages. The knitted wire which serves as separating steps between the agitator stages is also of Monel. In production use columns usually are used for a specific and continuing operation.

However, in case it is desired to clean a column, either to use it for other production steps, or to remove accumulated dirt, the column is cleaned either by simple draining, and if excessive dirt is collected at the interface, the whole column can be flushed from bottom or top by flushing with an excess of solvent or by stopping the flow of one of solvent phases.

Here are 15 TYPICAL APPLICATIONS for FIN-FAN AIR-COOLED Heat Exchanger Equipment

For many years, Fin-Fan Air-Cooled Heat Exchangers have proved their worth by giving reliable, economical service in many and varied applications. The Fluor Corporation, Ltd., and Griscom-Russell Company, pioneer-partners in the air-cooled heat transfer field, offer the following to show the large variety of practical applications for air-cooled heat transfer equipment and examples of relative size and duty of such equipment.



GAS COOLING	Natural Gas After-Cooler	13,920,000	80	46' x 25'
	Manufactured Gas Cooler	11,165,000	50	23' x 21'
	Natural Gas After-Cooler	3,400,000	15	11' x 21'
	Natural Gas Inter-Cooler	7,890,000	40	23' x 21'
VAPOR CONDENSING	Reflux Condenser	18,407,000	90	48' x 25'
	Primary Condenser	4,652,000	40	11' x 25'
	Still O.H. Condenser	4,875,000	34	16' x 21'
	Debutanizer Condenser	11,930,000	60	23' x 25'
	Still Reflux Condenser	39,110,000	60	46' x 25'
	Steam Condenser	87,300,000	300	138' x 31'
	Steam Condenser	2,880,000	10	10' x 6'
LIQUID COOLING	Vegetable Oil Cooler	628,000	15	6' x 17'
	Absorption Oil Cooler	14,915,000	100	32' x 17'
	Quenching Oil Cooler	1,665,000	50	23' x 25'
	Amine Solution Cooler	19,300,000	175	38' x 25'
	Stabilized Crude Cooler	125,100,000	700	315' x 25'
	Jacket Water	2,400,000	5.5	16' x 12'
	Jacket Water	28,900,000	100	70' x 24'

A new 8-page bulletin on Fluor-GR Fin-Fan Heat Exchangers is now being prepared. Write in and reserve your copy by post card or letter.

Why Fluor-GR Fin-Fans Lead The Field

The Fin-Fan Air-Cooled Heat Exchanger is unique in its development and manufacture. It is the result of the engineering skill of *two* companies, *each* long recognized as leaders in their respective fields. It combines Fluor's experience in the design, fabrication and field erection of air-moving systems with Griscom-Russell's experience in heat transfer.

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HEAT TRANSFER SYMPOSIUM—ATLANTIC CITY, DEC. 2-5, 1951

Division A of the A.I.Ch.E. Program Committee is organizing a symposium on heat transfer for the annual meeting in Atlantic City. Several outstanding papers on heat transfer are already on the horizon, and Prof. D. A. Katz and A. S. Foust of the department of chemical engineering, University of Michigan, Ann Arbor, Mich., who are planning the symposium, would like to obtain a few additional papers.

In addition to the program on heat transfer, the program committee is hopeful that it can adopt a new procedure which would involve preprinting papers before the meeting and limiting the meeting to a discussion of the papers. Decision as to whether this new plan can be followed cannot be made definitely at the moment and it is possible that the symposium will follow the usual pattern of paper presentations followed by brief discussions. In the new plan a five-minute review of the contents or salient points in a paper would be given to those in attendance, and this would be followed by the discussion. It is expected that preprints of the papers will be available some three weeks prior to the meeting, and, of course, the papers would be available at the time of registration at the meeting.

Manuscripts for the symposium in Atlantic City on heat transfer must be in the hands of the Program Committee by Aug. 15. Two copies of the paper should be sent to Professor Katz or Professor Foust in Ann Arbor and three copies sent to the editor of Chemical Engineering Progress. A copy of the letter of transmittal should be sent to Frank J. Smith, chairman, Technical Program Committee, Atlantic City, at the Pan American Refining Corp., 122 E. 42nd Street, New York 17. N. Y.

Division A of the Program Committee will review papers submitted and evaluate all papers before placing them on the program. In no case will a paper be accepted for the symposium if a manuscript is not available prior to Sept. 1. Minor revisions may be made in the manuscript as late as Oct. 1 in case the new plan of preparing preprints is followed.

OFFICERS RESEARCH UNIT OF A.S.E.E.

Three engineering research administrators were elected officers of the research unit of the American Society for Engineering Education. The three who became officers of the Engineering College Research Council on July 1 are Morrough P. O'Brien, chairman of the department of engineering at the University of California, Berkeley, made vice-chairman of the Council; A. A. Jakkula, executive director of the Research Foundation at Texas A & M College, and Eric A. Walker, director of the Ordnance Research laboratory at Pennsylvania State College and executive secretary of the Research and Development Board in Washington, both to be directors of the organization.

These selections were announced at the annual dinner of the research group in the Union building of Michigan State College by Gerald A. Rosselot, director of the Engineering Experiment Station at Georgia Institute of Technology and chairman of the Engineering College Research Council.

ALLOY STEELS NOW WORKABLE WHILE HOT

The use of cerium to improve the hot workability of high alloy corrosion-resistant heat-resisting steels was announced by the Carpenter Steel Company. The invention which applies to ferrous alloys containing nickel and one or more of the elements chromium, molybdenum, cobalt, copper, tungsten, silicon, manganese, columbium and vanadium, now enables these alloys to be forged and rolled into sheets, rods and tubes. Heretofore, these alloys could be cast satisfactorily but hot working would tear and crack the bars so that they were unusable.

The Carpenter Steel Co. claims combinations of cerium and lanthanum separately or in combination may be used within certain limits. The combinations are found in misch metal, the major part of which consists of cerium and lanthanum.

Shown above are the results of a cone test for hot forgeability. Test consists of casting a cone of the metal to be tested. Cone is heated to selected forging temperature and upset or hammered into a flat "pancake" as shown. Measure of forgeability is visual. Test can be used to determine the optimum amount of cerium to be used in a particular grade of steel, and also to determine the best range of forging temperatures. The two pancakes shown are of the exact same analysis . . . except that the one on the left, relatively free of cracks and tears, contains cerium.

ENGINEER SHORTAGE GRAVE—KILLIAN

Warning of "the grave shortage of scientists and engineers which we face in this country at the present time," Dr. James R. Killian, Jr., President of the Massachusetts Institute of Technology, told more than one thousand alumni at their annual dinner that this shortage will become still more acute in the next few years.

"This shortage," he said, "came about because of the unfortunate estimates given national distribution by the Bureau of Labor Statistics several years ago. These estimates at that time indicated that the country was faced with a surplus of engineers and the report had an obvious effect on the number of young men going into engineering schools.

"Last year," Dr. Killian said, "the engineering schools of the country graduated about 50,000 young engineers. This, of course, was an exceptionally high number, reflecting the wave of G.I.'s which had been going through the colleges. Based upon present enrollments, estimates have been made which indicate that in 1954 we will turn out of our engineering schools from 12,000 to 16,000 engineering students. This compares to an output from our engineering schools just before World War II of some 20,000 engineers a year.

"Because, therefore, of a decreased enrollment in the entering class this year and the prospect of a similarly small freshman class next year, we face the possibility of turning out fewer engineers than we educated before the war. . . . The country's demand for engineers is greatly in excess of what it was before the war. Estimates now place the annual demand around 30,000.

"This year the total number of engineers graduating will be on the order of 38,000, but of this total industry can count on getting only about half. According to a study recently made by the Engineering Manpower Commission of the Engineers Joint Council, industry will have a deficit of about 11,000 engineers this June. While industry will get half of the number graduating this year, estimates indicate that they will get only one fourth their needs next year. Again this does not take into consideration the need for engineers and scientists on the part of military establishments or other government operations.

"Our own experience points up these overall figures.

"M.I.T. could have placed this June three or four times as many graduates as we had available for jobs," he said.

(More News on page 28)

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MARGINAL NOTES

News of Books of Interest to Chemical Engineers

A Worthy Text for Teacher and Practician

Fluid and Particle Mechanics. C. E. Lapple (assisted by G. E. Alves, O. P. Bergelin, D. F. Boucher, H. L. Bunker, M. G. Kershaw, M. S. Malesson and R. D. Moore). (paper bound) (1951) v + 353 pp. $6.00.

Reviewed by J. M. Dalla Valle, Professor of Chemical Engineering, Georgia Institute of Technology, Atlanta, Ga.

THIS text constitutes an important addition to the chemical engineering literature. Considering that it is a compilation of published papers and classroom material, nevertheless a surprising degree of continuity of subject matter is dealt with. Mr. Lapple states that the aim of the publication is "to provide industrial and academic organizations with a readily usable critical review of recent developments in the field of Fluid and Particle Mechanics." This objective is admirably achieved, not only as regards competent discussion of many specialized topics, but also by means of well-chosen examples demonstrating techniques of computation.

There are 15 chapters in the text, 11 on fluid mechanics and four on particle technology. Chapter 1 deals with mass, energy and momentum balance and sets forth the fundamental equations of fluid flow. This is followed by a brief outline of turbulence theory applied to flow in pipes and across infinite plates. It is to be regretted in this connection that an indication of mass- and heat-transfer processes is not included, although this does not in any way diminish the value of the chapter. The next chapter (Chapter 3) on velocity head concept in pressure-drop theory is straightforward and contains practical information.

The flow of compressible fluids is discussed in Chapter 4. Herein Professor Lapple has done an excellent job. The chapter is concise, yet comprehensive and rich in numerical examples.

Ejectors have assumed an increasing degree of importance, yet there are few texts which deal adequately with the subject. Chapter 5, by four contributors, contains basic material and should be welcomed by the chemical engineering profession which has yet to utilize ejectors in many unit operations simply because pertinent and readily accessible information regarding them has been lacking. This chapter helps to remedy the existing situation.

Chapter 6 on two-phase flow in pipes presents an up-to-date discussion of contributions by Martinelli, Bergelin and Gazely. This is a rich field for further research since the information available is limited and because there are so few cases in chemical engineering practice where single-phase flow alone is involved. Chapter 7 on non-Newtonian flow is informative, but is perhaps oversimplified and if anything too condensed. Rheological flow is a subject of considerable complexity and deserves fuller discussion than is given in the chapter. Nevertheless, the inclusion of the fundamental properties of rheological flow, does make the engineer aware of the importance of subject.

The remaining chapters on fluid flow, Chapters 8-10 inclusive, are concerned with fluid metering, pipe-line design and pumps. This material is more or less of "handbook" type and leaves much to be desired. On the other hand, it does round out the objectives of the text and if omitted would leave a gap which many would criticize. Chapter 11 is on ventilation and includes all necessary design information. It is perhaps the best condensation of the subject the reviewer has seen and is recommended.

The chapters on particle technology cover general aspects pertinent to the field of chemical engineering. The authors have succeeded in bringing together a vast amount of useful information. Unfortunately, the omission of Professor Lapple's important contribution (with C. B. Shepherd) on particle dynamics is keenly felt. Chapter 14, on the collection of dust and mist, discusses the important types of commercial collectors used in industry and gives data on their applications and limitations.

The last chapter, dealing with fluidized beds contains many important relationships and represents the status of fluidization theory at this time.

The subject matter is indexed. The reviewer considers the text well suited for teaching purposes. There are numerous well-chosen examples at the end of each chapter, but over and above these, there is a need for a definitive work in fluid flow for chemical engineers. Professor Lapple and his associates have made a valuable contribution in this respect. The book is recommended to practicing chemical engineers.

Theory and Practical Details Advantageously Combined

Chemical Engineering for Production Supervision. David E. Pierce (Second Edition), McGraw-Hill Book Co., New York, N. Y. (1950) 290 pp. $4.00.

Reviewed by W. Hirschkind, Director of Research, Great Western Division, Dow Chemical Co., Pittsburg, Calif.

A SECOND edition within eight years speaks well for any technical volume.

The book is an excellent reference text for plant supervisors, foremen, and chief operators. Its study will result in a basic understanding of unit operations of chemical engineering by men without any formal technical education. The author has stressed description of operations rather than design of process equipment, for which the book is in no way intended. It should be useful for a training program for operating personnel.

It is clearly written and contains considerable detail of a practical nature not usually found in the standard chemical engineering texts.

The first two chapters, which cover general principles and transformation of energy, are good reading for persons with little technical training. The discussions are short and to the point and are amply illustrated by examples and problems. The following chapters dealing with unit operations are considerably more advanced, and would seem to require some technical background which most persons confronted with technical problems either have, or readily acquire.

Generally, each chapter discusses the basic theory to some extent and then presents examples of equipment and of solving simple problems. A good balance has been maintained between brevity and a more thorough but more complex coverage.

This book, like its first edition, should find considerable usefulness in the field for which it is intended.

Books Received

Quantum Mechanics of Particles and Wave Fields. Arthur March. John Wiley & Sons, Inc., New York, N. Y. (1951) 292 pp. $5.50.

Preparation of Organic Intermediates. David A. Shirley. John Wiley & Sons, Inc., New York, N. Y. (1951) 328 pp. $6.00.

"KARBATE" BRAND IMPERVIOUS GRAPHITE HEAT EXCHANGERS *FOR ALL PURPOSES!*

The "Karbate" impervious graphite heat exchangers illustrated are used extensively as boilers, coolers, condensers, vaporizers, evaporators, heaters and absorbers in handling corrosive chemicals, either hot or cold. They *all* can be had in a complete size range. *All of them* offer the following advantages:

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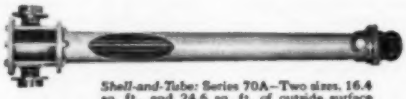
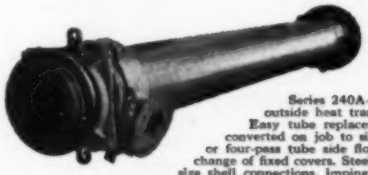


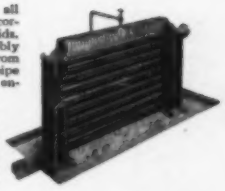
Shell-and-Tube: Series 70A—Two sizes, 16.4 sq. ft., and 24.6 sq. ft. of outside surface area respectively. Tubes easily replaced in the field. Interchangeable single and double-pass construction.

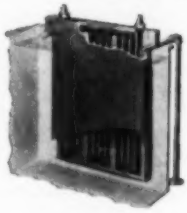
Style FH heat exchangers (Size 1½): Are especially recommended for tanks with solution depths of 16 inches or less. Write for catalog section S-6750.



Series 240A—70.6 feet of outside heat transfer surface. Easy tube replacement. Easily converted on job to single, double, or four-pass tube side flow by simple change of fixed covers. Steel, shell, oversize shell connections, impingement plates and drain and vent plugs integral with shell end castings. Stainless-steel baffles assembled with steel tie rods to form protective cage for tube bundle. Removable "Karbate" tube bundle. Write for catalog sections for S-6690, S-6715 for details of applications, maintenance, sizes and characteristics of these exchangers.

Cascade coolers: For all cooling jobs involving corrosive gases and liquids. Complete cooler assembly may be made quickly from 4 standard items in 5 pipe sizes. Capacity easily enlarged or reduced by adding or subtracting standard sections. Compact construction to save plant room. No special supporting structure needed. Write for catalog section S-6780.





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CANDIDATES FOR MEMBERSHIP IN A. I. Ch. E.

The following is a list of candidates for the designated grades of membership in A.I.Ch.E. recommended for election by the Committee on Admissions.

These names are listed in accordance with Article III, Section 7, of the Constitution of A.I.Ch.E. which states:

Election to membership shall be by vote of the Council upon recommendation of the Committee on Admissions. The names of all applicants who have been approved as candidates by the Committee on Admissions, other than those of applicants for Student membership, shall be listed in an official publication of the Institute. If no objection is received in writing by the Secretary within thirty days after the mailing date of the publication, they may be declared elected by vote of Council. If an objection to the election of any candidate is received by the Secretary within the period specified, said objection shall be referred to the Committee on Admissions, which shall investigate the cause for such objection, holding all communications in confidence, and make recommendations to the Council regarding the candidate.

Objections to the election of any of these candidates from Active Members will receive careful consideration if received before *August 15, 1951*, at the Office of the Secretary, American Institute of Chemical Engineers, 120 East 41st St., New York 17, N. Y.

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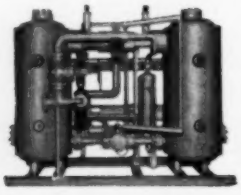
(*Continued on page 34*)



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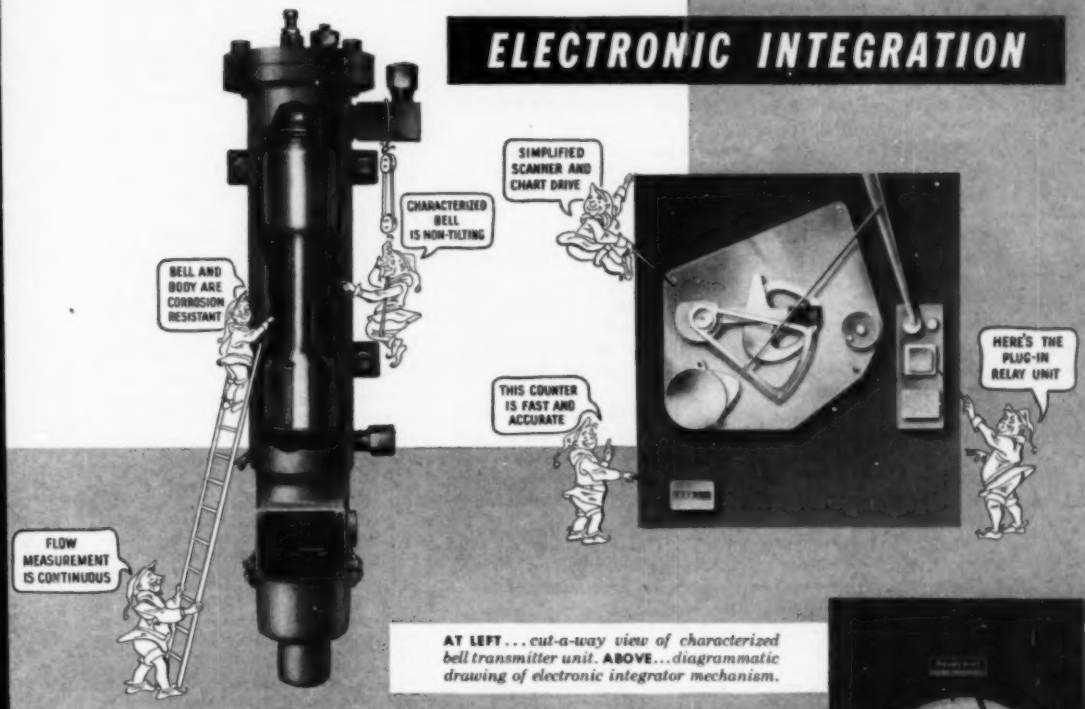
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It uses Niagara No-Frost Liquid Spray to keep frost and ice from ever forming. It gives you, automatically, refrigeration with no defrosting, and full capacity NEVER cut down by ice building up progressively on refrigeration coils.

Now, a NEW design No-Frost Liquid concentrator, using a new principle, takes away moisture as fast as it is condensed by evaporating it at low temperature — not boiling it away at high temperature. It has 14 times the capacity of the old method per dollar of investment — one concentrator will handle a battery of high capacity spray coolers.

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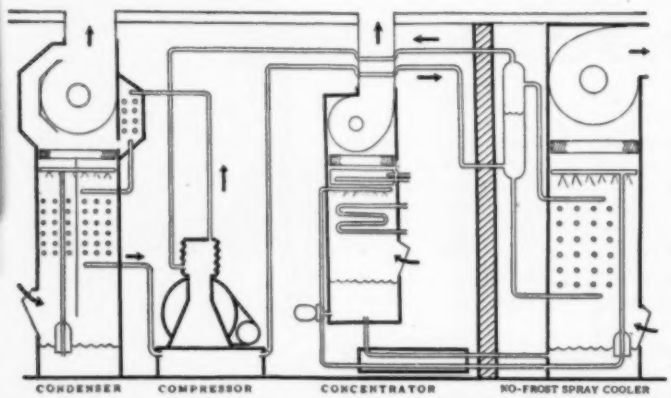
The extra capacity and lower cost both for equipment and operating makes this method advantageous for every type of refrigeration use — both for freezing and for moderate temperatures — for large "live" loads as in meat chilling or in fruit and vegetable pre-cooling — for rooms that are filled and emptied of product daily, such as milk rooms and terminal storage warehouses.

You get true trouble-free refrigeration . . . No brine . . . no salt solution . . . no dirt . . . no mess . . . It is entirely clean; you get rid of dirt and odors. You reduce both equipment and operating costs.

For complete information write to the Niagara Blower Company, Dept. EP 405 Lexington Avenue, New York 17, New York.



A simple method, easily maintained. Saves a third of your refrigeration cost. Ask for Niagara Bulletins 118 and 119.

(*News continued from page 22*)

SULFUR SHORTAGE DUE TO FOREIGN LAG

U. S. production of sulfur is more than ample to meet all of the needs of American industry, but the demands of our allies for this strategic mineral are causing a serious shortage, according to Langbourne M. Williams, Jr., president of Freeport Sulphur Company.

"Our production of this basic raw material is two and a half times prewar levels," he stated. "We are doing everything we can to boost output still further, but a continuing shortage is inevitable as long as we are called upon to fill the global demands that are being made upon us."

Brimstone from the salt dome deposits of the Gulf Coast, the major U. S. form, accounts for nearly half the world output. Other forms are pyrites, sour natural gas, petroleum refinery gas, smelter gas and gypsum. World production from all sources in 1950 contained an estimated 11,700,000 tons of sulfur.

A number of new projects are under way in the United States, Mr. Williams explained, but such additional production as may be achieved will not solve the shortage unless other countries rely much more heavily upon the alternate sulfur sources.

"The cost of obtaining sulfur from these other sources, however, is considerably higher than the cost of brimstone," he said. "Our brimstone sells in the domestic market at $22 per ton—less than a penny per pound—compared with prices for foreign sulfur ranging as high as $120 a ton.

"Because of the wide disparity in price, and the fact that our brimstone not only is cheap but is a better product, the world has preferred to save dollars by dipping into our resources," he added. "Many of these countries have access to supplies of sulfur in one form or another that are more than adequate for their needs.

"Although in the past these served as the historic source of sulfur for these countries, they are being utilized to a relatively slight extent because of their higher cost. If these countries were to make full utilization of their reserves, it would not be necessary for them to make such unprecedented demands upon our own brimstone production.

"An intensive effort is being made to boost our brimstone production. Three new mining plants are being constructed in Louisiana and Texas, but because of the unpredictable and highly speculative nature of sulfur mining, the amount they will add to production cannot be determined until after they are in actual operation. In addition, 13 projects are under way to obtain sulfur from other sources.

"On the other hand, production from some of the older brimstone mines is bound to decline. Therefore, unless some rich new deposit of brimstone is discovered, the long-term solution to the shortage must be achieved through further expansion of production from higher-cost sources of sulfur. Fortunately, the nation's reserves of such sulfur are vast."

The three new brimstone mines now being developed are Spindletop dome of Texas Gulf Sulphur Company near Beaumont, Texas, and Starks dome of Jefferson Lake Sulphur Company in Louisiana, both of which are expected to be in production this year, and the Bay Ste. Elaine dome of Freeport scheduled to begin operation near the end of 1952.

The Bay Ste. Elaine project, lying near the Louisiana coast, is an amphibious operation. The entire plant, capable of supplying nearly 2,000,000 gallons of superheated water a day to melt the sulfur, will be built on barges at Grande Ecaille, 75 miles distant by water from the dome, floated to the mine site and sunk in place. Because the land over the dome is unstable—two thirds of the area is under water—the molten sulfur will have to be transported in special barges to the company's storage facilities at Port Sulphur.

In addition to the Bay Ste. Elaine project, Freeport is actively pursuing a long-range prospecting program, Mr. Williams said. An exploratory drilling program is now in progress at domes in Louisiana and Texas.

LECTURES TO HONOR McCORMACK-FREUD

An annual lectureship to honor the first first two men to head departments of chemistry and chemical engineering at Illinois Institute of Technology, Chicago, was recently established by Omicron chapter of Phi Lambda Upsilon, honorary chemical society. The men honored are Professor Emeritus Harry McCormack and Benjamin B. Freud. The first McCormack-Freud honorary lecture, entitled "Development of Chemistry and Chemical Engineering at Illinois Institute of Technology," was delivered April 25.

Professor McCormack was professor of chemical engineering at the Institute from 1907 to 1946 and was a consulting chemical engineer. Dr. Freud taught chemistry at the college from 1904 to 1949 and was chairman of the chemistry department from 1937 to 1947.

NYLON FOR JAPAN

Toyo Rayon Company, Ltd., of Tokyo, has been licensed to manufacture nylon in Japan under patents of E. I. du Pont de Nemours & Company, it was announced early in June. The agreement with the Toyo firm has been approved by the Japanese government and the Supreme Commander of Allied Powers.

Toyo Rayon, which is manufacturing nylon on a modest scale at present, has plans for extensive operations under the agreement. Nylon is being produced by Toyo under the trade-mark Amilan.

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DEVELOPMENT PROGRAM ANNOUNCED BY U. D. & C.

An inter-industry cooperation program aimed at furthering chemical product research and development was announced last month by the United Dye & Chemical Corp. The aim of the program will be to offer the facilities and experience of the company to those who have chemical problems or who need assistance in the development of practical chemical ideas.

In presenting the new program, David S. Fischman, president of United Dye & Chemical, stated, "We believe there are many individuals and small companies who have practical chemical products and processes but lack the research equipment, the concentration of technical personnel, and the 'know-how' to develop their ideas to commercial status. We have the laboratories, the research equipment, and the testing equipment that these firms and individuals need. We have the technical personnel and 153 years in the chemical industry. . . . ."

The company has set up a Research and Development Service, and this group will evaluate the ideas brought to its attention. Development will be on a mutual basis.

United Dye & Chemical has a long experience in the organic chemistry field, Mr. Fischman pointed out, particularly in vegetable and synthetic dyes and pigments. Plants operated by the company's subsidiaries are located in Belleville, N. J., and Chester, Pa., in the United States and in Great Britain, France, British West Indies, Corsica, Italy, Belgium and Haiti.

MORE NITROGEN FOR SOLVAY

Plans to expand synthetic nitrogen capacity by 100,000 tons per year, together with conversion to natural gas from coke as a source of hydrogen, at a total cost of more than $25,000,000, were announced last month by The Solvay Process Division, Allied Chemical & Dye Corp., New York, N. Y. The expansion will take place during the next two years at Solvay's Hopewell (Va.) and South Point (Ohio) nitrogen plants.

The Hopewell plant was built in 1928 and is the largest of its kind in the United States. The South Point plant was built and operated by Solvay for the Government during World War II and was purchased by the company in 1946. Since 1946, the aggregate annual capacity of the Hopewell and South Point plants has been expanded by 80,000 tons of nitrogen to supply the increasing needs for nitrogen products.

(*More News on page 32*)

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Drugstore ... hospital ... pharmaceutical laboratory ... wherever prescriptions are filled or proprietary remedies are formulated, you'll find versatile U.S.P. Glycerine at work.

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*By Government authorities responsible for inventory control under the National Defense Act.

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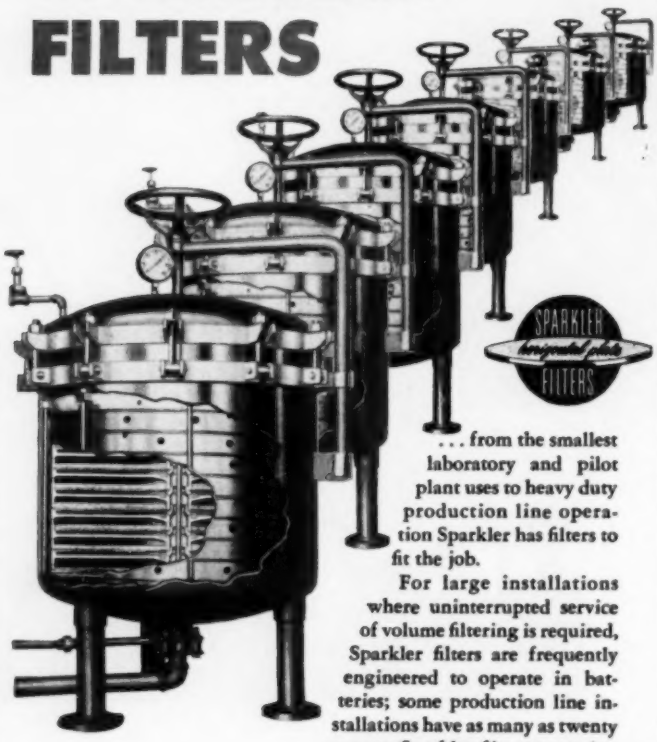

. . . from the smallest laboratory and pilot plant uses to heavy duty production line operation Sparkler has filters to fit the job.

For large installations where uninterrupted service of volume filtering is required, Sparkler filters are frequently engineered to operate in batteries; some production line installations have as many as twenty or more Sparkler filters operating together. Sparkler filters are made in a complete range of metals, stainless steel, mild steel, rubber lined, etc., steam jacketed and brine jacketed.

STANDARD MODELS ARE AVAILABLE MADE WITH THESE MECHANICAL SPECIFICATIONS:

Plate diameter sizes 8", 13½", 18", 33"
Filter area in one filter unit 1 sq. ft. to 200 sq. ft.
Cake space capacity 1 lb. to 300 lbs. of filter aid.
Flow rates from 10 G. P. H. to 48000 G. P. H. in one unit depending on viscosity of liquor.

For personal service and engineering consultation on your particular filtering problem, write Mr. Eric Anderson.

SPARKLER MANUFACTURING COMPANY
MUNDELEIN, ILL.

(*News continued from page 30*)

Robert C. Swain, vice-president in charge of research and development, American Cyanamid Co., was recently elected honorary chairman, American Section, Society of Chemical Industry, for 1951-52. Dr. Swain joined the research staff of American Cyanamid Co. in 1934 and advanced by stages to become vice-president in 1946. He is also a director of the Jefferson Chemical Co. He was educated at Leland Stanford University and received the Ph.D. degree from the University of Berlin in 1932.

RECORD OF SAFETY BY CHEMICAL PLANTS

Chemical plants are among the safest places in the country in which to work, according to figures released recently by the Manufacturing Chemists' Association, Inc.

A record low of 4.56 injuries per million man-hours worked was achieved in 1950 by the Association's member companies, which account for about 90% of total U. S. chemical production.

"This accident rate is less than half the national average," Dr. A. G. Cranch, chairman of the Association's safety committee said, "and lower than that of any other industry except communications."

Chemical injury rates have been substantially below the average for all industry for many years, according to Dr. Cranch, "but chemical plants have made especially notable progress in reducing industrial injuries since 1946." The average rate for the Association's member companies has dropped 40% during the past five years. Dr. Cranch attributes this record to improved safety measures adopted in the industry and to good cooperation by employees in observing safety rules and precautions.

The collection and dissemination of statistics on chemical plant safety has been an activity of the Association's general safety committee for a number of years. This committee is also actively engaged in keeping the industry informed of latest safety techniques. With the assistance of the Association's medical advisory committee it has prepared 39 chemical safety data sheets. These are used by industry throughout the world as authentic sources of information on how to handle chemicals safely and what to do in the way of first aid and medical treatment in case of accident.

AMENDMENTS TO N. Y. REGISTRATION LAW

The Professional Legislation Committee of the A.I.Ch.E., under the chairmanship of John M. Weiss, reports that several significant amendments have been enacted to the Engineers Registration Law in New York State. These may be of interest to those who have registered in the State or who are considering registering.

According to the chairman, an amendment has been made which enables the New York State Board to accept the examinations passed by engineers in training in another state. This means they may be certified as engineers in training in New York State without re-examination.

Mr. Weiss reports that this amendment also legalizes a practice of the New York State Board which accepted examinations passed in other states, as a basis for waiving all or part of the prescribed professional engineering examinations in New York.

A second feature is that the law requiring licensed professional engineers and land surveyors to register with the county clerk has been repealed. One effect of this repeal is that nonresident engineers licensed in New York State, may complete their registration now and be listed in the roster, without establishing an office address in New York State.

The third point is that the fee for a professional engineer's license has been reduced from $25 to $20 and the fee for renewal registration has been increased from $2.00 to $5.00. This renewal is a biennial requirement.

SULFUR EXTRACTION PLANT IN WEST TEXAS

Phillips Chemical Co. will start construction of a large sulfur extraction plant in West Texas, according to an announcement made by K. S. Adams, chairman, and Paul Endacott, president, of both Phillips Chemical Co. and its parent, Phillips Petroleum Co.

This plant is designed to extract from natural gas nearly a quarter of a million pounds of elemental sulfur per day and will be located in the Permian Basin oil fields near Goldsmith. The output will be used by the company in connection with its ammonium fertilizer plant at Adams Terminal near Houston. The plant will manufacture sulfur from the hydrogen sulfide rich vent gas from residue gas-treating operations in the Goldsmith area. A variation of the Claus process will be used.

The Ralph M. Parsons Construction Co. will erect the plant which will be completed in the first quarter of 1952.

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The importance of modern pH equipment is widely recognized in many fields . . . from food processing to sewage treatment—mining and smelting to electroplating—textile manufacturing to sugar refining. But when considering pH equipment for your problems always remember this . . .

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Only Beckman provides such a wide variety of versatile, accurate and dependable electrodes—a type to meet virtually every industrial, research, laboratory and medical requirement. This is your assurance that, in Beckman pH equipment, you have maximum adaptability to widely-varying pH applications.

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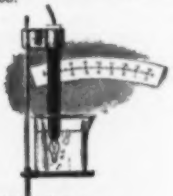
Where abrasive slurries or rough industrial service are problems, Beckman has the answer with electrodes so strong, so tough, so chemically-resistant to alkalies and acids they set entirely new standards of durability!

Extreme Temperatures

Want to make measurements in solutions as cold as 20°C. *below* zero . . . or as hot as 130°C. *above*? Hot, cold or in-between, there's a Beckman pH Electrode to do the job!



Wide pH Range

Electrodes covering the full pH range permit accurate measurements even at extreme limits with negligible sodium ion error whether test solution is hot or cold!

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CHEMICAL ENGINEERS OF GREATER NEW YORK HONOR BLASE OF PFIZER

Second from left is Edwin W. Blase, Pfizer Co. chemical engineer, winner of the Achievement Award of the Chemical Engineers of Greater New York. With him are three members of the awards committee, left to right, Joseph A. O'Connor, of Chemical Engineering; the chairman, William Trotter, of Celanese Corp., and Robert D. Burleson, of Merck and Co. The award, open only to chemical engineers under 32, was presented June 7. Mr. Blase is 27. He was cited as "sole inventor of the Q-process for the recovery of terramycin which expedited the early large-scale production of a life-saving drug." Terramycin was first isolated by Pfizer researchers in the summer of 1949 and, by March, 1950, was available in quantity to the medical profession.

CRITERIA OFFERED TO DRAFT BOARDS BY E. J. C.

An Engineering Manpower Commission Newsletter dated June 8, 1951, was mailed recently to the 3800-odd local draft boards throughout the country on "Industrial Internship — A National Necessity." The purpose of this Newsletter is to aid members of these draft boards in considering requests for deferment based upon participation in industrial training. It sets up four questions which may be used as criteria for judging such deferment cases. These questions are as follows:

1. Is the program restricted to graduate engineers, or the equivalent?
2. Does it teach advanced fundamentals at the graduate level?
3. Does it integrate the trainee into the particular branch of essential industry so that he may make his maximum contribution to the national effort?
4. Is it well established in the organization or is it an expedient adopted since the latest national emergency?

If the answers to such questions are yes, then the individual engineer is a registrant whose case merits occupational deferment in the national health, safety and interest.

Members of the editorial committee of the Engineering Manpower Commission of E.J.C. which provided the Newsletter are:

S. C. Hollister, chairman, dean of the college of engineering and vice-president Cornell University, Ithaca, N. Y.

H. N. Muller, assistant to vice-president in charge of engineering, Westinghouse Electric Corp., East Pittsburgh, Pa.

M. H. Trytten, director, Office of Scientific Personnel, National Research Council, 2104 Constitution Avenue, Washington, D. C.

Judging by reports from the draft boards reaction to this letter has been favorable.

JOINT TITANIUM RESEARCH ANNOUNCED

A joint research venture toward the development of processes for the production of titanium metal was announced by Charles Allen Thomas, president of Monsanto Chemical Co., and Richard S. Morse, president of National Research Corp., Cambridge, Mass.

Work initiated by National Research Corp. will be greatly expanded under terms of the agreement, and additional work will be carried on at Monsanto's central research department at Dayton, Ohio. The project will be under the direction of Robert A. Stauffer, vice-president and technical director of National Research Corp., and N. N. T. Samaras, Monsanto's central research department director.

PATENTS RELEASED BY A.E.C. FOR PUBLIC USE

Descriptions of 26 patents owned by the U. S. Government and held by the Atomic Energy Commission have been transmitted to the U. S. Patent office. They will be registered and listed in the official register of patents.

As part of its program to make nonsecret technological information available for industry's use the Commission will grant nonexclusive, royalty-free licenses on the listed patents. A total of 284 Commission-held patents and patent applications has now been released for licensing.

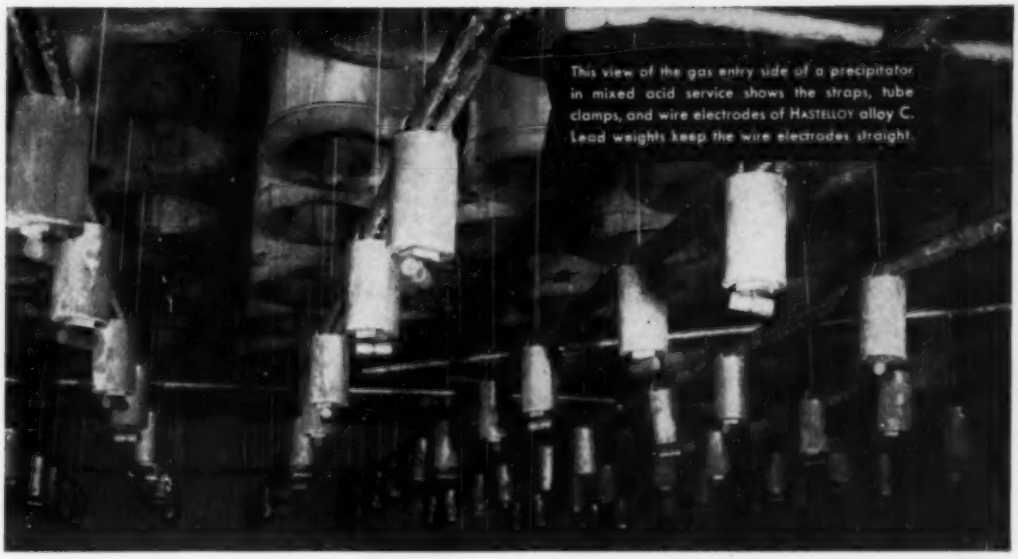
Applicants for licenses should apply to the chief, patent branch, Office of the General Counsel, U. S. Atomic Energy Commission, Washington 25, D. C., identifying the subject matter by patent number and title.

(*More News on page 36*)

CANDIDATES FOR MEMBERSHIP

(*Continued from page 26*)

William E. Green, *Norwalk, Calif.*
Frank E. Guptill, Jr., *Whittier, Calif.*
Robert J. Gustafson, *Rochester, N. Y.*
William S. Haines, *Webster Groves, Mo.*
Gardner L. Hayward, *Boston, Mass.*
Joseph T. Hogan, *New Orleans, La.*
R. Robert Hollibaugh, *Bonner Springs, Kan.*
William G. Hudson, *Westfield, N. J.*
Lawrence Isakoff, *Baldwin, N. Y.*
Louis Jablansky, *New York, N. Y.*
Elmer D. Jones, *Morristown, N. J.*
Milton Klein, *Chicago, Ill.*
L. Randall Koenig, *Webster Groves, Mo.*
Martin A. Kroesche, *Houston, Tex.*
Shiu Lee, *Berkeley, Calif.*
Arthur E. Lindroos, *River Edge, N. J.*
James N. Malina, *Pasadena, Calif.*
Robert V. Maresca, *New York, N. Y.*
Julian B. McFarland, Jr., *Edgewater Park, N. J.*
Hobart C. McGinnis, *St. Albans, W. Va.*
James Donald Miller, *Long Beach, Calif.*
Calvin Q. Morrison, *Albany, Ore.*
Ralph H. Nielsen, *Corvallis, Ore.*
Virgil Orr, *Shreveport, La.*
Fred R. Pence, *Wilmington, Del.*
Aloysius J. Polaneczky, *Philadelphia, Pa.*
John A. Rauscher, *Kirkwood, Mo.*
William L. Richards, *Fort Worth, Tex.*
James B. Robinson, Jr., *Louisville, Ky.*
Harry J. Ruland, *Brooklyn, N. Y.*
Joan A. Schilk, *State College, Pa.*
William Schotte, *New York, N. Y.*
Harold A. Schulte, Jr., *Detroit, Mich.*
Donald W. Schurch, *Aruba, N. W. I.*
Norman C. Sidebotham, *Drexel Hill, Pa.*
Karl E. Sieber, *Corning, N. Y.*
John A. Tantillo, *Wilmington, Del.*
Dalton T. Taylor, *Winfield, W. Va.*
Edward D. Thomas, *Pittsburgh, Pa.*
Myron G. Urdea, *Dearborn, Mich.*
Hal P. Van Fossen, *Rifle, Col.*
Gustave J. Weiss, *New York, N. Y.*
William L. Welch, *Ponca City, Okla.*
Forrest S. Wilcox, *Clifton Heights, Pa.*



This view of the gas entry side of a precipitator in mixed acid service shows the straps, tube clamps, and wire electrodes of HASTELLOY alloy C. Lead weights keep the wire electrodes straight.

Precipitators Improved...

WITH NICKEL-ALLOY ELECTRODES

Electrostatic precipitators for removing corrosive liquids and solid particles from industrial process gases give better and longer service, without maintenance, when HASTELLOY alloy wire is used for electrodes. The alloy has excellent mechanical strength (130,000 psi) and is not attacked by the corrosive agents. This combination of properties makes it possible to use electrodes that are only 0.073 in. in diameter. The effective diameter of the lead-covered wire formerly used in most units was more than 0.500 inch. The smaller cross-section of the HASTELLOY alloy wire permits the use of higher voltages with a more uniform corona pattern.

HASTELLOY alloy C electrodes have been installed in mist precipitators where hydrochloric, sulphuric, sulphurous, and mixed acids were present. A recent check on an installation in an acid plant showed that the alloy C wires experienced no weight loss after a year's operation. Lead-covered steel wires, on the other hand, lasted an average of 11 months with electrode failure at least once a week.

For information on wrought forms of HASTELLOY alloy, write for the booklet, "HAYNES Wrought Alloys Price List."

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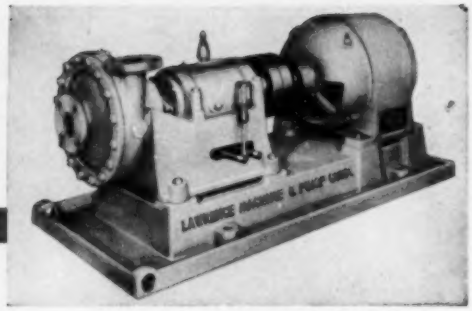


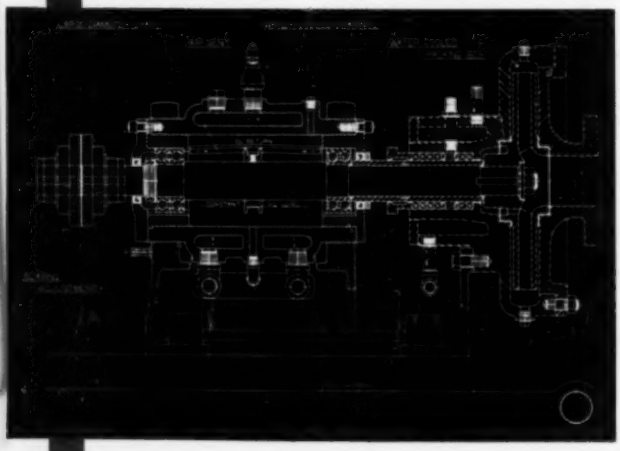
Lawrence Process Pump

PROCESS PUMPS for HANDLING LIQUIDS at HIGH TEMPERATURE and HIGH PRESSURE

The pumping of corrosive and/or abrasive liquids at high temperatures and pressures requires design techniques beyond the capabilities of most pump manufacturers. Lawrence engineers have specialized in this difficult field for over 80 years and can offer you the benefits of their broad experience.

Typical of Lawrence advanced engineering is the process pump illustrated, made from corrosion-resistant metals with packing box and bearing housing both water cooled. Note also the clean-cut appearance and accessibility of the principal components.

If you have a difficult pumping problem, write us the pertinent details. No obligation.



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INTERNATIONAL MINERALS ACQUIRES INNIS, SPEIDEN

International Minerals & Chemical Corp. will acquire the assets of Innis, Speiden & Co., manufacturers of potash compounds, as well as gums, waxes and insecticides. The action to acquire Innis, Speiden & Co. came last month when stockholders authorized the board of directors to increase the number of International Minerals & Chemical common shares from 2 million to 2½ million, and to issue an exchange of unissued shares of common stock.

Innis, Speiden & Co. was founded in 1816, and now operates a manufacturing plant at Niagara Falls, N. Y., a refining and processing plant in Jersey City, N. J., and a warehouse in Chicago, Ill. F. Eberstadt & Co. acted as financial advisers to Innis, Speiden & Co.

LIBERAL EDUCATION IN ENGINEERING OFFERED

Twelve selected liberal arts colleges have entered into an agreement with Columbia University's School of Engineering whereby the country's need for more broadly trained engineers can be served. Under this plan, after the satisfactory completion of three years' study of liberal arts at one of the cooperating colleges, the student can be automatically admitted to Columbia for two years of engineering study. Completion of the five-year program will result in appropriate Bachelor's degrees from both Columbia and the liberal arts college. The five-year combined program for training engineers will begin operating in September with the entering classes of freshmen at the liberal arts college.

The new five-year plan recognizes that the professional engineer, in addition to being technically competent in his own field, should have a clear understanding of the relationship between his technology and the social, economic and political life of the nation. Under this plan, the student has a unique opportunity for finding himself, since during his liberal arts course, he can change his choice of career and the engineering school is given the opportunity to concentrate its efforts upon giving an outstanding engineering education to a group of highly selected students.

THODE HEADS C.I.

One of Canada's outstanding nuclear chemists, Dr. H. G. Thode, M.B.E., of Hamilton, Ont., was elected President of The Chemical Institute of Canada for 1951-52. Dr. Thode is Principal of Hamilton College of McMaster University and is also head of the University's Department of Chemistry and Director of Research there.

SECRETARY'S REPORT

S. L. TYLER

THE Executive Committee met at the offices of the Institute on June 8, 1951. The first items of business were approval of Minutes of the previous Executive Committee Meeting, Treasurer's report and bills for the month. All those applicants for membership whose names were listed in Chemical Engineering Progress, May, 1951, issue, were elected to the grades of membership as indicated. In addition to the above 51 applicants were elected to Student membership.

J. W. Eldridge was appointed counselor of the student chapter at the University of Virginia to succeed W. R. Winsbro, and Richard L. Ferm was appointed counselor of the student chapter at the University of New Mexico to succeed H. G. Hoover.

John H. Barnard, Jr., and Virgil R. Bonnette, Junior members, were added to the Suspense List because of their having entered the Armed Forces.

The following changes were made in the personnel of the Local Sections Committee: appointment of E. A. Gastrock (New Orleans) replacing D. L. Stockbridge, E. C. Mirus (Western New York) and C. O. Buttrill (Sabine Area). Mrs. Mary Bailey was added to the Membership Committee representing the Detroit Section and J. W. Axelson was appointed to replace D. D. McLaren of the New Jersey Section.

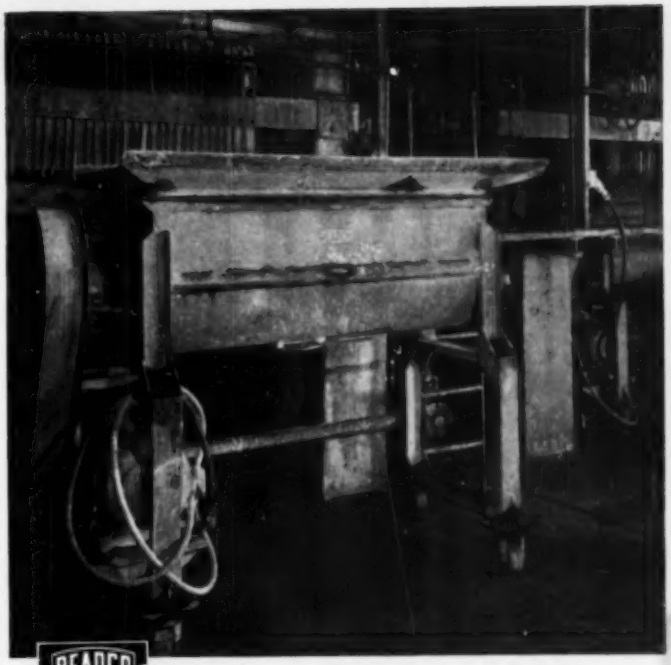
Nine resignations from membership were received and accepted. Four elections to Junior Membership were rescinded because of non-acceptance.

The Finance Committee had discussed at some length the question of a proper financial surplus which the Institute should attempt to establish and it was recommended to the Executive Committee that every effort be made to maintain a surplus equivalent to about one year's operations as this would then insure continuity of operations for a reasonable period in time of difficulties.

Publications Available from Secretary's Office

Letter Symbols for Chemical Engineering ($1.00) Accepted and published by the American Standards Association as American Standard Z10.12—1946.

Chemical Engineering Problems (1946) ($1.00) Contains a total of 469 problems in the following categories: Stoichiometry—Gas Mixtures, Thermochemistry, Combustion of Gaseous Fuels, Combustion of Liquid Fuels, Combustion of Solid Fuels; Kinetics—Applied Chemical Kinetics; Unit Operations—Fluid Flow, Heat Transfer, Evaporation, Distillation, Filtration, Humidity and Air Conditioning, Drying, Dif-



READCO CHEMICAL EQUIPMENT

READCO Mixers facilitate FILTER CAKE HANDLING

Searching for a means of eliminating expensive, time consuming methods of handling tough, unmanageable filter cake, the manager of a large processing plant consulted Read Standard engineers.

After thorough testing and analysis of his process, Readco Spiral Ribbon Mixers were recommended and installed.

Filter cake, which was formerly scraped from the press and laboriously shoveled into drums, is now dropped into mixers where it is quickly reduced to the proper consistency.

The homogeneous, semi-liquid mass is now poured from Readco Mixers into drums ready for shipment, greatly reducing the length of processing time necessary.

Read Standard's engineering specialists in the design and application of processing equipment are available to assist you in determining the proper equipment to fit your special needs.

Read Standard manufactures a complete line of mixing, blending, sifting and material handling equipment. *Read Standard Corporation, Bakery-Chemical Division, York, Pennsylvania.*

ANOTHER SPECIAL PROCESSING PROBLEM SOLVED BY READ STANDARD

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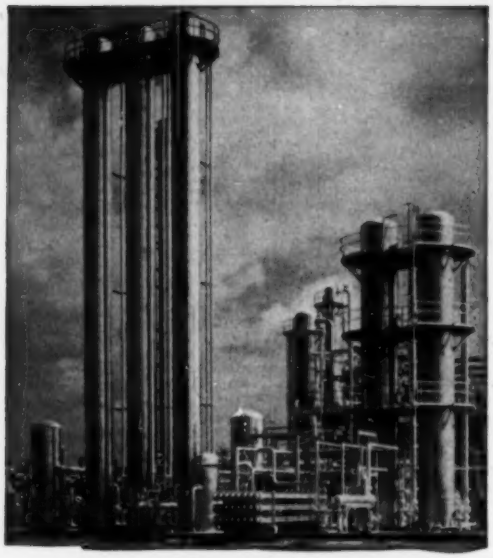

Southwest Gas Producing Co.—Uses

FLORITE

The recently completed gasoline plant of Southwest Gas Producing Company at Dubach, La., gathers wet gas from the surrounding area and employs new highly efficient methods for separating its ethane and propane components. Ninety million cubic feet of natural gas is handled daily, yielding a daily output of 230,000 gallons of liquefied hydrocarbons. The Fish Engineering Corporation of Houston designed and constructed this model plant.

In the two towers at the right of our illustration, wet gas is dehydrated with Florite and then passed to the three towers at the left for high-pressure absorption.

Floridin Products, adapted to a wide range of advanced industrial and technical uses, include several specially prepared forms of . . . . . .

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SECRETARY'S REPORT

(*Continued from page 37*)

fusion, Absorption, Extraction, Crushing and Grinding, Size Separation, Washing and Decantation.

Student Contest Problems and The Prize Winning Solutions (1932-1949) ($3.00) This volume contains the Contest Problems and the First Prize Winning Solutions since the inception of the Contest. In addition to the above, individual copies of the Problem and the First Prize Winning Solution for the years 1947, 1948 and 1949 are also available at $1.00 each.

The Engineering Profession in Transition (1946) ($.50) This survey was prepared by a special survey committee of Engineers Joint Council's Committee on the Economic Status of the Engineer. It contains data in graphical and tabular form on the distribution of trained engineers by fields, and by industries, and reports on median salaries based on years of experience; 94 pages.

Symposium on Relationship Between Pilot-Scale and Commercial Chemical Engineering Equipment—Preprint, White Sulphur Springs, W. Va., meeting (March 11-14, 1951) ($2.50) Contains nine papers presented at White Sulphur Springs.

IT'S BEEN PUBLISHED

Every so often an item published in the past, comes to the editor's attention which has a bearing and an interest for the present. Through the lack of publicity, these publications may not have come to the attention of members interested in the particular sphere of engineering that they cover; therefore, we are including this news item in Chemical Engineering Progress as a reminder and perhaps a first notice to our members.

In 1938, A. Dexter Hinckley, then assistant to the engineering dean of Columbia University and now secretary of the Illuminating Engineering Society, prepared for the Committee for Professional Recognition of the E.C.P.D. a report on the study of the qualification procedures in the fields of accounting, architecture, law, and medicine. The purpose of this was to determine and analyze the current methods of granting recognition to candidates in the several professions of law, medicine, architecture and accounting, and the study embraces all the United States, and where possible, the development of a process of accrediting candidates.

A brief summary of this was reported at the time and has not come to the attention of too many of the engineers and it shows via a table, the size of the professional group according to the U. S. census of 1930, and the numbers ordinarily qualified for admission to professions. These statistical data might be outmoded at present but the practices are essentially the same and for 15 cents you can obtain a copy of this summary from E.C.P.D., 29 West 39th St., New York 18.

LOCAL SECTION

SOUTHERN CALIFORNIA

The June 19 meeting was held at the Roger Young Auditorium in Los Angeles with W. B. Shanley, Los Angeles representative of Universal Oil Products as featured guest speaker on "Aromatics by Platforming."

This section held its monthly dinner meeting at Scully's Cafe, in Los Angeles May 15. Some 85 guests and members were present to hear a discussion by Willard Geer, assistant professor of physics, University of Southern California, on "Industrial Applications of Television." Dr. Geer is noted for his development of one of the four methods of color television transmission and reception. He presented the advantages and disadvantages of each system together with some of the uses so far developed and suggestions for future applications.

The local section welcomed some Senior members to the meeting—J. G. Dean, consultant, who has been a member of A.I.Ch.E. since 1908 and Walter Schmidt, president and general manager of Western Precipitation Corp., who has been with the Institute since 1915.

Reported by Gale S. Peterson

TIDEWATER VIRGINIA CHEMICAL ENGINEERS CLUB

This Club visited the Hopewell plant of the Hercules Powder Co.'s Virginia cellulose department May 26. This was the second in the Club's series of plant trips. The Club members also inspected the department's chlorine plant and research and control laboratories.

Reported by Daniel Post

RHODE ISLAND SOCIETY OF CHEMICAL ENGINEERS

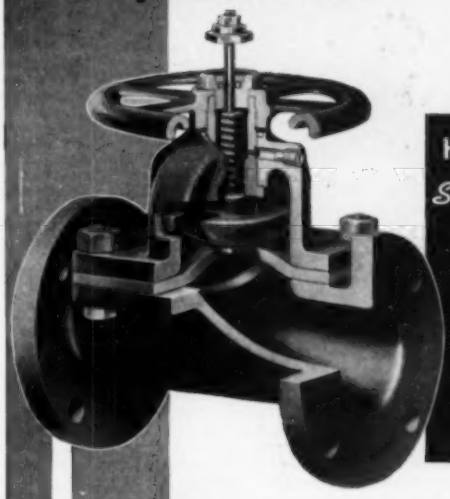
The final meeting of the year was held May 23, 1951, at Johnson's Hummocks Restaurant in Providence. The speaker was Raymond W. Jacoby, branch manager of the Providence office of the Ciba Co., who spoke on "Recent Developments in Textile Finishing."

Officers for next year are:

Chairman—Karl A. Holst, director of research, Rumford Chemical Co.
Vice-chairman—Harold E. Graves, chairman, department chemical engineering, University of Rhode Island.
Secretary-Treasurer—Robert W. Nelson, American Reinforced Paper Co.
Director—Herman B. Goldstein, manager, production and development, Warwick Chemical Co.
Saul Ricklin (retiring chairman)

Reported by Warren L. Towle

Here is the valve that revolutionized flow control of Sulphuric Acid . . .



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Sulphuric acid up to 66° Bé can now be valved without leakage, dripping or sticking . . . with Hills-McCanna Diaphragm Valves equipped with the new L-1 plastic diaphragms. This new development combines the tried and proved Saunders Patent pinch clamp principle with a diaphragm of a special type of plastic that is particularly resistant to concentrated sulphuric acid.

Hills-McCanna Diaphragm Valves with L-1 diaphragms are suitable for temperatures up to 125° F. and pressures up to 100 psi. Sizes from ½" thru 4" are available, handwheel operated, quick opening (lever operated) and sliding stem models. Choice of nearly 50 body materials or linings including cast iron, cast steel, Durimet or glass lined.

Literature is available describing Hills-McCanna Valves with data on their use for acid and other services. Write for your copy outlining the nature of your application. HILLS-McCANNA COMPANY, 2438 W. Nelson St., Chicago 18, Ill.

The valve illustrated in cut-a-way above is the Hills-McCanna Model 6600 semi-sealed bonnet acid valve with L-1 plastic diaphragm and tell-tale travel stop indicator. Valves of this type can be furnished where particularly rigid safety precautions must be taken.

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PRESENT AND PAST OFFICERS OF CHARLESTON (W. VA.) SECTION

In back row, left to right: M. C. Guthrie, past secretary, Du Pont Co.; J. H. Howell, past treasurer, Carbide and Carbon Chemicals Co.; G. K. S. Connolly, secretary, Carbide and Carbon Chemicals Co., and D. J. Porter, chairman, past chairman, Westvaco Chemical division.

In front row, left to right: A. B. Stiles, past chairman, Du Pont Co.; R. E. Soden, treasurer, Monsanto Chemical Co., and R. Voorhees, vice-chairman, Carbide and Carbon Chemicals Co.

Officers not present: J. E. Crawley, Jr., member-at-large, Du Pont Co., and C. C. Richiusa, past member-at-large, Monsanto Chemical Co.

SABINE AREA

This section recently concluded its first year as a local section of A.I.Ch.E. with a report of an active year which began in October, 1950. Subsequent meetings were held in November, February, 1951, April, 1951, and on May 25 at Beaumont, Tex., there was a social meeting. The speaker at that get-together W. M. Casey addressed 58 people on, "Is America's Freedom or International Peace our Objective?"

The following officers have been elected for the year 1951-52:

Chairman—Scott Reeburgh, Gulf Oil Corp.
Chairman-elect—W. H. Litchfield, Gulf Oil Corp.
Secretary-Treasurer—D. J. McCarthy, Texas Co.
Executive Committee—F. A. Beverly, Magnolia Petroleum Co.; J. C. Covington, Du Pont Co.; D. T. Parks, Gulf Oil Corp.
Retiring Chairman—Carroll O. Buttrill, Magnolia Petroleum Co.

Reported by W. J. Burich

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OHIO VALLEY

At the final meeting of the year, held June 4, 1951, an inspection trip was made of the Schoenling Brewing Co., Cincinnati, Ohio.

At the May 7 meeting graduate students from the University of Cincinnati presented papers in the technical session held at the Herman Schneider Foundation Building, Cincinnati. George D. Moon gave a paper on "The Effect of Surface Active Agents in Liquid-Liquid Extraction." The second paper was delivered by Richard F. Kayser entitled, "Problems in Obtaining Adequate Kinetic Data for Vapor Phase Catalytic Reactions."

The following officers were elected for the 1951-52 fiscal year:

Chairman.....Ray Katzen
Vice-Chairman.....Art Greber
Treasurer.....Ted Wiehe
Secretary.....Stan Baechle

Reported by A. C. Greber

SOUTH TEXAS

Approximately 120 members and guests of this Section visited the Point Comfort Works of the Aluminum Company of America at Port Lavaca, Tex., May 18. This represented the largest attendance the Section has had at a regular monthly meeting in several years. After an inspection trip through the plant members and friends were guests of the Aluminum Company for refreshments and dinner.

The evening program consisted of the showing of a film "This Is Aluminum," a talk by G. R. Stout, works manager, concerning the over-all activities of the Aluminum Company, and an interesting speech on "Aluminum Chemicals" was given by J. W. Newsome, chief of chemicals division of Aluminum Research located in East St. Louis.

Plans are underway for the Sixth Annual Technical Session to be held at Galveston, Oct. 19, 1951. Dr. W. B. Franklin is serving as general chairman for this all-day session.

Reported by Henry D. Foster

ST. LOUIS

An inspection trip to the A. E. Staley Manufacturing Co., Decatur, Ill., constituted the May 12 meeting of this Section. The trip started with an orientation lecture during which mimeographed leaflets and flow sheets were distributed to 39 attending members. The remainder of the morning was spent in the monosodium glutamate unit and the pilot plant. After lunch at the cafeteria the afternoon was spent in the starch plant.

A. T. Pickens

AKRON

A meeting was held May 10, 1951, at Knight Hall at the University of Akron, which began with an inspection trip of the chemical and physical testing laboratories of Knight Hall. The tour provided an opportunity to see a modern chemistry building containing mill, curing, and testing rooms with the latest equipment for rubber research and study. The speaker of the evening W. L. Davidson, director, physical research department, B. F. Goodrich Co., talked on "Atomic Energy in Peace and War."

At a previous meeting, 65 members and guests heard a talk by W. I. Burt, vice-president of manufacturing of the B. F. Goodrich Chemical Co. and vice-president of A.I.Ch.E., in which he gave brief descriptions of the functions and duties of the various committees of the Institute.

Henry H. Sheperd of the Bird Machine Co., South Walpole, Mass., spoke on Filtration and the facilities and filter equipment available by Bird for performance and cost-conscious engineers who, he said, can take advantage of a combination of Bird's experience in the field and then complete testing, research, and development facilities.

Reported by Edmund Duplaga

BOSTON

Ichthyologists

At the May 11 meeting, Dick Morse, president and founder of National Research Corp., gave a talk on "Launching New Enterprises." In developing this topic, he described in detail the required essentials for getting a new enterprise under way. These essentials, he believed, included an idea, an organization or team and capital.

At a previous meeting Stanley Lovell, president of Lovell Chemical Co., Waterford, Mass., talked on "Undercover Engineering." Dr. Lovell, former OSS director of research and development, was asked innumerable questions. His speech was screened for security reasons.

The Annual Ladies Night was held June 1 at the Cambridge Yacht Club. Present were 130 members and their wives.

Reported by R. Antonsen and H. Avery

CLEVELAND

A meeting of this Section was held May 22, 1951, at the research laboratories of the Standard Oil Co. of Ohio. An inspection trip through these facilities was followed by dinner. Following the dinner, movies entitled, "Industries of Ohio, and Waterways of Ohio," were shown.

Reported by G. H. Metzger

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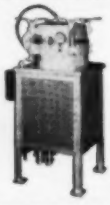

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FUTURE MEETINGS AND SYMPOSIA OF A.I.Ch.E.

Chairman of the A.I.Ch.E. Program Committee

Walter E. Lobo, The M. W. Kellogg Co., 225 Broadway, New York 7, N. Y.

MEETINGS

Rochester, N. Y., Sheraton Hotel, Sept. 16-19. 1951.

Technical Program Chairman: D. E. Pierce, General Aniline & Film Corp., 230 Park Ave., New York, N. Y.

Annual—Atlantic City, N. J., Chalfonte-Haddon Hall Hotel, Dec. 2-5, 1951.

Technical Program Chairman: Frank J. Smith, Pan American Petroleum & Transport Co., 122 East 42nd St., New York 17, N. Y.

Atlanta, Ga., Atlanta Biltmore Hotel, Feb. 17-20, 1952.

Technical Program Chairman: H. E. O'Connell, Ethyl Corp., Box 341, Baton Rouge, La.

French Lick, Ind., French Lick Springs Hotel, May 11-14, 1952.

Technical Program Chairman: W. W. Kraft, The Lummus Co., 385 Madison Avenue, New York, N. Y.

Chicago, Ill., Palmer House, Sept. 4-6, 1952.

Technical Program Chairman: D. A. Dahlstrom, Chem. Eng. Dept., Northwestern University, Evanston, Ill.

Annual—Cleveland, Ohio, Hotel Cleveland and Carter Hotel, Dec. 7-10, 1952.

Technical Program Chairman: R. L. Savage, Dept. Chem. Eng., Case Inst. of Tech., Cleveland 6, Ohio.

Annual—St. Louis, Mo., Dec. 13-16, 1953.

SYMPOSIA

Maintenance

Chairman: D. E. Pierce, General Aniline & Film Corp., 230 Park Ave., New York, N. Y.
Meeting—Rochester, N. Y.

Industrial Applications of Photography

Chairman: Carl Gath, Eastman Kodak Co., Kodak Park Works, Rochester, N. Y.
Meeting—Rochester, N. Y.

Round Table Discussion of Chemical Engineering Education

Chairman: W. E. Gift, Tennessee Eastman Corp., Kingsport, Tenn.
Meeting—Rochester, N. Y.

Opportunities in Sales for Chemical Engineers

Chairman: F. J. Curtis, Monsanto Chem. Co., 75 Rust Bldg., 1001 15th St. N.W., Washington 5, D.C.
Meeting—Atlantic City, N. J.

Chemical Engineering Fundamentals

Chairman: W. C. Edmister, Chem. Eng. Dept., Carnegie Inst. of Tech., Pittsburgh 13, Pa.
Meeting—Atlantic City, N. J.

Effective Speaking

Chairman: L. P. Scoville, Jefferson Chem. Co., 711 Fifth Ave., New York 22, N. Y.
Meeting—Atlantic City, N. J.

Vacuum Engineering

Chairman: W. W. Kraft, The Lummus Co., 385 Madison Avenue, New York, N. Y.
Meeting—French Lick, Ind.

Authors wishing to present papers at a scheduled meeting of the A.I.Ch.E. should first query the Chairman of the A.I.Ch.E. Program Committee, Walter E. Lobo, with a carbon copy of the letter to the Technical Program Chairman of the meeting at which the author wishes to present the paper. Another carbon should go to the Editor, F. J. Van Antwerpen, 120 East 41st Street, New York 17, N. Y. If the paper is suitable for a symposium, a carbon of the letter should go to the Chairman of the Symposia, instead of the Chairman of the Technical Program, since symposia are not scheduled for any meeting until they are complete and approved by the national Program Committee. Before authors begin their manuscripts they should obtain from the meeting Chairman a copy of the Guide to Authors, and a copy of the Guide to Speakers. The first book covers the preparation of manuscripts, and the second covers the proper presentation of papers at A.I.Ch.E. meetings. Presentations of papers are judged at every meeting and an award is made to the speaker who delivers his paper in the best manner. Winners are announced in *Chemical Engineering Progress*, and a scroll is presented to the winning author at a meeting of his local section. Since five copies of the manuscript must be prepared, one should be sent to the Chairman of the symposium and one to the Technical Program Chairman of the meeting, or two to the Technical Program Chairman if no symposium is involved and the other three copies should be sent to the Editor's office. Manuscripts not received 70 days before a meeting cannot be considered.

PEOPLE

Edward W. Comings will become head of the school of chemical and metallurgical engineering at Purdue University, Lafayette, Ind., beginning September 1, 1951, succeeding R. Norris Shreve. Dr. Comings was educated at the University of Illinois where he graduated with high honors. He then went to Massachusetts Institute of Technology where he received the D.Sc. degree. After spending several years as a chemical engineer with the Texas Co., Beacon, N. Y., he started teaching first at North Carolina State College in Raleigh, then going to the University of Illinois in 1936 as assistant professor where he advanced through the various ranks to become full professor in 1947. He was official investigator for the Office of Scientific Research and Development and assistant director, Munitions Development Laboratory at the University of Illinois. His research interests include such chemical engineering subjects as liquid extraction and high pressure measurements. He contributed a chapter on Filtration to "The Applications of Chemical Engineering," by Harry McCormack (ed.). He is no stranger to the Purdue Campus as he taught there for three summers.

Professor Comings succeeds **R. Norris Shreve,** a member of the engineering faculty of Purdue University since 1930 who will resume his professorship of chemical engineering. He will devote his time to his teaching, professional contacts, research and writing. Professor Shreve went to Purdue University after more than 20 years of engineering and industrial experience in designing, building and operating plants in the chemical industry. While at Purdue, Professor Shreve has written four books as author or co-author and some 70 technical articles.

Edward R. Weidlein, formerly director of the Mellon Institute of Industrial Research, University of Pittsburgh, Pittsburgh, Pa., is now president. This change is in accord with new bylaws recently adopted at the annual meeting of the board of trustees. Dr. Weidlein has also been elected chairman of the board. As in the past, when he was director, Dr. Weidlein continues to be the chief executive officer of the corporation and as previously is in charge of the active management of all its affairs.

CHANGES AT MONSANTO ST. LOUIS PLANT

Robert E. Lenz recently became assistant to the director of the general engineering department, Monsanto Chemical Co. Mr. Lenz joined the company's Alabama plant in 1940 as a research chemical engineer, becoming plant engineer in its Dayton (Ohio) rocket propellent plant in 1942. In 1945 he became a chemical engineer in the company's general development department at St. Louis and subsequently when the process engineering section of that department was transferred to the general engineering department, he became its assistant manager.

Clyde P. Orr, who succeeded Mr. Lenz as assistant manager of the process engineering section, joined the company in 1942 as a chemical engineer at the Merrimac division, Everett, Mass. In 1946 he transferred to the general development department at St. Louis, and subsequently was named project engineer in 1949.

William F. Scanlan, a graduate chemical engineer of Purdue and former assistant chief engineer on a Navy troop transport, has joined the staff of the Houston (Tex.) district sales office of the Whiting Corp. His four years' experience at the Swenson's Illinois plant in Harvey included engineering, sales and operating assignments in both the chemical and pulp and paper divisions.

J. Gordon Mitchell, chemical engineer of the Cumberland (Md.) plant of the Canadian Chemical Co. Ltd., has become plant engineer of the Edmonton (Alta.) operation according to an announcement by Harold Blancke, president of Celanese Corporation of America. He has been with the company since 1929.

James Tucker MacKenzie was recently awarded the 1951 Herty Medal for "outstanding contributions to chemistry in the Southeast" by the Georgia section of the American Chemical Society. Dr. MacKenzie is technical director of the American Cast Iron Pipe Co., Birmingham, Ala.

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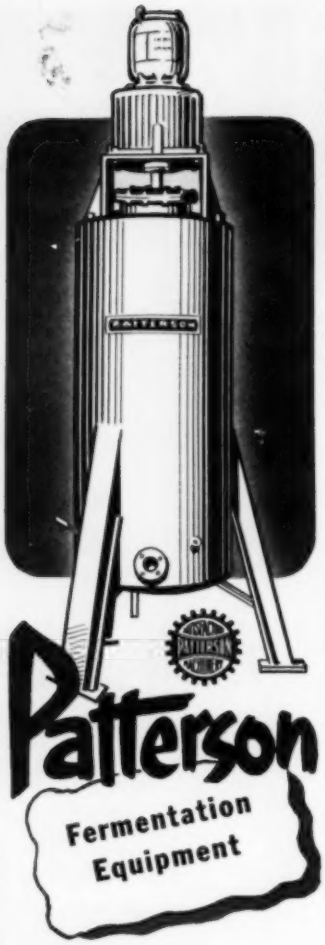

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MONTREAL

J. J. O'Neill will head the new paint development laboratory within the chemical division of the Goodyear Tire & Rubber Co. Mr. O'Neill has had 10 years' experience as research and development chemist in the paint industry and is the holder of several patents on the development of petroleum resins. He holds a degree in chemical engineering from Drexel Institute of Technology, Philadelphia, Pa. He will devote most of his time to further refinements for Pliolite S-5, the company's synthetic material which is used in the paint industry in the formulation of chemical resistant finishes, stucco, aluminum finishes, etc.

Carl S. Carlson, a research chemist with the Standard Oil Development Co., Elizabeth, N. J., is the new national vice-president of Phi Lambda Upsilon, honorary chemical society. Early in his career Dr. Carlson was assistant professor of chemical engineering at Louisiana State University and later transferred to the University of Pennsylvania. Subsequently he was associated with the Shell Oil Co., and in 1945 he joined the Standard Oil Development Co.

I. M. LeBaron, research engineer with International Minerals & Chemical Corp. since 1942, has been appointed director of the corporation's research laboratories. With offices in Chicago and in Mulberry, Fla., Dr. LeBaron will continue to report directly to the vice-president in charge of research and will aid him in special assignments. Dr. LeBaron, inventor or co-inventor of various patents, held teaching fellowships at Rhode Island State College, New York University, Louisiana State University and the Colorado School of Mines. He received a Dr. Eng. from Colorado School of Mines in 1941 and from that year to 1942 he was research engineer for the Aluminum Company of America at its research laboratories in New Kensington, Pa. With Aluminum Company of America he worked on the development of high strength aluminum alloys. Since joining International he has been responsible for many developments among them a process for concentration of manganese ores.

Bernard R. Sarchet has been named manager of Koppers' Kobuta plant at Monaca, Pa., and will also be responsible for the operation of the adjacent government butadiene plant, having been formerly in charge of its reactivation. Mr. Sarchet received his Master's degree in chemical engineering from the University of Delaware, joining the Koppers Co. as an operator in its engineering and construction division in 1941. He was subsequently general foreman of the company's butadiene division at Kobuta; supervising engineer in its research and chemical engineering department; and manager of the chemical division at the Oil City plant.

Gaur Gopal Modak is a chemical engineer in training with the Consolidated Mining & Smelting Co., Ltd., Trail (B.C.) Canada. Prior to this assignment he was chemical engineer with Pfaudler Co., Rochester, N. Y.

Richard L. Marion has been appointed research chemist of the Sun Rubber Co., Barberton, Ohio. He will be primarily concerned with coordinating the work of Sun's laboratory with manufacturing operations in the development of new processes and improvement of existing techniques. Marion goes to Sun from Fenn College in Cleveland, where he served as instructor in chemical engineering for the past four years. He holds a B.S. degree in chemical engineering from Purdue University and has completed six years of graduate study at Buffalo University and Case Institute of Technology. His background includes two years as senior research engineer with the National Aniline division of Allied Chemical & Dye Corp., Buffalo, N. Y.

H. L. TRUMBULL RETIRES

Harlan L. Trumbull, who in 1947 was named by the American Chemical Society as one of the ten "ablest chemists and chemical engineers" working in the American rubber industry, has retired after 32 years with the B. F. Goodrich Co. Dr. Trumbull worked on many major government assignments during World War II, in which period he made two inspection tours of German synthetic rubber operations. During World War I he helped to establish the U. S. Army's Chemical Warfare Service. Dr. Trumbull is credited with the achievement of making latex out of rubber. He received his Bachelor's and Master's degrees in chemistry at Univ. Washington.

Edward G. Locke, chief of the forest utilization service in the Pacific Northwest Forest and Range Experiment Station is being transferred from Portland to the U. S. Forest Products Laboratory in Madison, Wis. He will be chief of the division of derived products, which conducts research in wood chemistry. Dr. Locke received a B.S. degree in chemical engineering in 1928 from Oregon State College and a Ph.D. in organic chemistry from Ohio State University in 1932. After employment as a chemist for the Ohio State Highway Commission and private firms, Dr. Locke returned to Oregon State College in 1936 as assistant professor of chemical engineering. He was a chemical engineer in the industrial and resource development division of Bonneville Power Administration from 1942 to 1944. He spent five months in 1945 in Europe primarily investigating the chemical utilization of wood in Germany.

John E. McKeen, president of Charles Pfizer & Co., Brooklyn, N. Y., was awarded the honorary degree of doctor of engineering by the Polytechnic Institute of Brooklyn at the 96th annual commencement held June 13 in the Brooklyn Academy of Music. Mr. McKeen entered the employ of Charles Pfizer & Co. as a control chemist upon his graduation from Polytechnic with the degree of chemical engineer.

George E. Holbrook, formerly manager of the plant development section of Du Pont's organic chemicals department, has been named assistant director of the company's development department. Dr. Holbrook has been associated with Du Pont since 1933, serving successively as research chemist; head of the new products division; and assistant director of the Jackson Laboratory, Deepwater Point, N. J. Following a short period as general superintendent of development of the company's Chamber Works, in 1949 he transferred to the Wilmington plant, where he served as assistant director of the organic chemicals department's technical division, then department engineer. Later he was named manager of the plant's development section.

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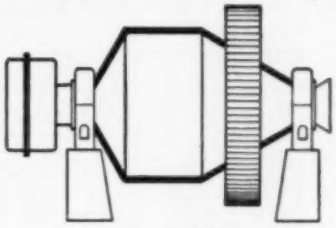

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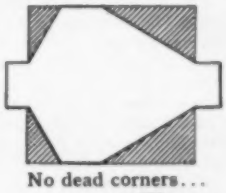

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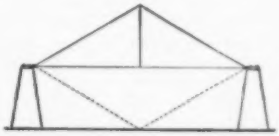

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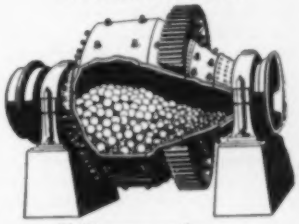

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CHILTON HONORED FOR SERVICE TO A.S.A.

Thomas H. Chilton, president of A.I.Ch. E. and technical director, development engineering division, engineering department, Du Pont Co., has been awarded a certificate of service by the American Standards Association in recognition of his work in the development of American standards. Dr. Chilton has just retired as a member of the A.S.A. Standards Council where he represented the American Institute of Chemical Engineers. More than 2,200 leading industrial firms are members of A.S.A. in addition to 110 national technical societies and trade associations.

K. Nagaraja Rao, a native of Bangalore, India, has gone to Indonesia under a six-month contract with that government, to aid in developing a technical training program for the small-scale workshop industries. This effort at promoting the standard of living and the national income will be under the joint financing of the E.C.A. technical assistance program and the Indonesian government. Rao, a chemical engineer with the Institute of Gas Technology, Chicago, received a Bachelor's and Master's degree from the University of Mysore, India, and worked for his Master's and Ph.D. degree in chemical engineering at the Illinois Institute of Technology.

Allen N. Smith has been promoted from assistant professor to associate professor of chemical engineering at the University of Louisville, Louisville, Ky. Dr. Smith is a graduate of Tulane University and was awarded a Master's degree in chemical engineering at Georgia School of Technology (1943) and a Ph.D. degree in chemical engineering at Oregon State College (1948). A member of several technical societies, he is a registered professional engineer in the state of Kentucky.

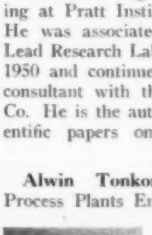
William E. Ranz has been appointed assistant professor of chemical engineering at the University of Illinois, Urbana, Ill. Dr. Ranz received his Ph.D. degree at the University of Wisconsin, and has been on the staff of the Engineering Experiment Station at Illinois for the past year.

John R. Bowman, head of the Mellon Institute's department of research in physical chemistry, concurrent with recent changes, has been added to the executive staff of the Institute.

Reuel C. Stratton, supervising chemical engineer of The Travelers Insurance Companies of Hartford, Conn., who for several years has been sole liaison representative between the life insurance industry and the United States Atomic Energy Commission, has recently been appointed a member of the hazards survey group of the Atomic Energy Commission.

Ralph H. McCormack has been appointed professor of chemical engineering at the University of Detroit. Dr. McCormack joined the Detroit staff May 1 to take part in the University's plans to expand the program in chemical engineering to include graduate study and to enlarge its research activities. A graduate of the University of Chicago, the University of Illinois and Virginia Polytechnic Institute, Dr. McCormack taught chemical engineering at Pratt Institute from 1937-1951. He was associated with the National Lead Research Laboratories from 1944-1950 and continues that association as consultant with the Baker Castor Oil Co. He is the author of numerous scientific papers on solvent extraction.

Alwin Tonkonogy, president of Process Plants Engineering Co., Newark, N. J., recently announced the opening of the company's new office in the Industrial Office Building, Broad Street, Newark. The expanding activities in the design and construction of chemical and process plants necessitated the move. Since 1932 Mr. Tonkonogy has been affiliated with several companies including the Industrial Gas Engineering Co., National Grape Corp., Industrial Process Engineers, and the Kellex Corp. He has been associated with the Process Plants Engineering since July, 1950. Mr. Tonkonogy received an M.S. in Heat Transfer Chemistry from the University of Tennessee.



(More About People on page 48)

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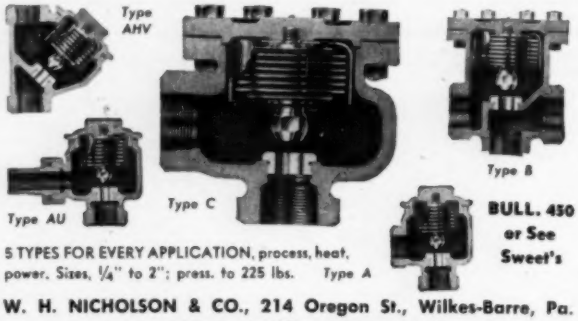

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PROCESS ENGINEER

Recent graduate or chemical engineer with one or two years' oil refinery experience desired. Letter should include details of education, experience, family and military status and salary expected. Personal interview can be arranged. Box 4-7.

SALES ENGINEER — For New York and Eastern territory by an Ohio manufacturer of process tanks, heat exchangers, and chemical-proof specialties. Must train several months at plant. Advise training and salary requirements. Box 5-7.

EMPLOYMENT OPPORTUNITY

CHEMICAL ENGINEER: M.S. or B.S. level, 3-5 years' experience desirable for work on high pressure boiler research in water chemistry and water-treating problems. Opportunity for advancement in well-equipped research department of reliable company. Location Northern Ohio. Box 6-7.

EMPLOYMENT OPPORTUNITY

CHEMICAL ENGINEER: B.S. or M.S. for work in research department of major equipment manufacturer. Three to five years' experience desirable in research on wood-pulping processes and development of equipment. Opportunity for advancement. Location Northern Ohio. Box 7-7.

YOUNG CHEMICAL ENGINEER—Well familiar with soda ash and paper and pulp plants design, urgently needed for well-paid and extremely promising assignment. Please write with detail of experience to Box 17-7.

PROFESSOR AND HEAD OF DEPARTMENT OF CHEMICAL ENGINEERING — Desired qualifications: Ph.D. in chemical engineering, teaching and industrial experience, special competence in chemical engineering thermodynamics.

ASSISTANT PROFESSOR OF CHEMICAL ENGINEERING — Desired qualifications: M.S. in chemical engineering, teaching and industrial experience.

Both of the above positions in a small endowed college of engineering located in metropolitan New York. Opportunities for consulting and research, but emphasis on undergraduate teaching. Standard academic responsibilities, privileges, and compensations. Applicants must be prepared to start September 6, 1951. Send full resume to Box 21-7.

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CHEMICAL ENGINEER—Now in academic work desire permanent industrial employment. Five years' industrial, ten years' teaching and consulting experience. Ph.D. degree, licensed. Special interest in equipment development and process design. Box 8-7.

CHEMICAL ENGINEER—M.Ch.E. Age 31. Supervisory experience in pilot plant design, construction, operation, and correlation of data. Experience in petroleum, heavy chemicals, and instrumentation. Desire responsible position. Minimum salary, $7500. Box 9-7.

PROJECT ENGINEER—M.Ch.E. Ten years' diversified design and production experience both organic and inorganic industry. Excellent pilot plant development development experience. Age 33, married. Desire executive-type work with future. Box 10-7.

CHEMICAL ENGINEER—B.Ch.E., 1948, married. Diversified experience in setting-up and trouble-shooting production lines in Government Arsenal. Desire position where chemical background and production experience can be united. Location immaterial. Box 11-7.

CHEMICAL ENGINEER — B.Ch.E., 1940. Eleven years' experience in research and development, chiefly organic processes. Desire responsible position in research and development. Location immaterial. Box 12-7.

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CHEMICAL ENGINEER—D.Eng. Fall 1951. Plant design, development, consulting, teaching experience. Strong background kinetics, thermodynamics, unit operations. Veteran, family, 34. Qualified for liaison; consultation, research, development, supervision. Box 14-7.

REGISTERED CHEMICAL ENGINEER—Extensive successful experience management medium-size inorganic chemical plants with organic and inorganic research and development departments. Seeking heaviest responsibilities. Box 15-7.

CHEMICAL ENGINEER PROFESSOR—M.I.T. Doctor, licensed engineer, A.I.Ch.E. Active member, nine years' research, development, process design and teaching experiences, is looking for more responsible position in industry or university. Specialty: unit operations, fluidization, thermodynamics, synthetic organic and heavy chemicals. Box 16-7.

CHEMICAL ENGINEER—B.S. Receive M. S. Sept. Two years varied experience in research and process development. Married, veteran, age 27. Desire position in development or process design. Prefer New York-New Jersey area, but will consider others. Available Sept. Box 18-7.

CHEMICAL ENGINEER—B.S., age 37. Fifteen years of intensive and successful work in organic chemicals, including process development, equipment design, and production management. Seek challenging position. Prefer eastern U. S. $10,000 minimum. Box 19-7.

CHEMICAL ENGINEER — B.Ch.E. Catholic Univ. of America, June, 1951. Age 27, single, draft exempt. Interested in production work, process design and technical sales. Location immaterial and willing to travel. Box 20-7.

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PEOPLE

(*Continued from page* 46)

Henry Avery has been appointed manager of the plasticizer division of the Pittsburgh Coke & Chemical Co. He will be responsible for directing and coordinating the division's expanding sales, production, technical service and development activities. He will be located in the central office of the company in the Grant Building, Pittsburgh, Pa.

Mr. Avery graduated from Massachusetts Institute of Technology in 1941 with a degree in chemical engineering. During World War II he served three and one-half years overseas.

While on the staff and faculty of the Ordnance Department overseas, Mr. Avery wrote many technical articles such as "Ordnance Lessons Learned During the Italian Campaign." He received the U. S. Legion of Merit and an Italian Government medal. In May, 1947, after a year at the Florida naval stores plant of G. L. Cabot, Inc., Mr. Avery was transferred to Cabot's Boston headquarters as plasticizer sales and product development engineer.

While in Boston he was secretary of the Boston Section of A.I.Ch.E.

Ben King Duffy, sales engineer, General American Transportation Corp., Chicago, Ill., has been appointed district sales manager of the plate and welding division for the Pittsburgh district of the company. Mr. Duffy will have charge of sales for such General American products as are fabricated by the company's plate and welding division, as well as Wiggins conservation structures and Wiggins gasholders.

Lyman Hall Allen, Jr., formerly with Foster D. Snell, Inc., has been appointed division engineer of the viscose section of the central engineering department of Celanese Corporation of America. He went to Snell late in 1950 from the American Viscose Corp., Philadelphia, Pa.

Henry E. Ford, who has been assistant director of Du Pont's development department since 1942, became director of the department June 1. Mr. Ford started with Du Pont in the explosives department immediately after his graduation from the University of Pennsylvania in 1922. He began as a chemist in the Eastern Laboratory. Subsequently he became supervisor of production at the Repauno Works, superintendent of power and later of acid production at the Barksdale (Wis.) works, later returning to Repauno as superintendent of acid production. He served Du Pont also in its London office as assistant manager and manager. He returned to Wilmington and entered the development department in 1939.

William T. Dickens has been appointed manager of the new Monsanto Chemical Co.'s plastics plant at Cincinnati, Ohio. He actively participated in the Cincinnati project since its inception last year. Mr. Dickens received his chemical engineering degree from Yale in 1938 and joined Monsanto that year as a research engineer. The next ten years, he was successively a design engineer in the general engineering department; area supervisor and later production superintendent in the Texas City (Tex.) plant, and general superintendent of the Lustrex styrene department in Springfield. Subsequently he was named plant manager, and later production representative for the new plant.

Wallace F. Armstrong has been appointed works manager of Ethyl Corp.'s new chemical plant now under construction in Houston, Tex. Mr. Armstrong, who received his M.S. in chemical engineering from the University of Florida, joined Ethyl in 1939 and has been supervisor of the ethyl chloride operations in Baton Rouge, La., for the past six years.

Necrology

C. E. SHAFER

Charles E. Shafer, Jr., shift supervisor of the General Electric Co., Richland, Wash., died recently. He had previously been associated with the Du Pont Co., in Old Hickory, Tenn., and at Oak Ridge, Tenn., and in Richland. In September, 1946, he accepted an appointment with the General Electric as shift supervisor on the Atomic Energy Commission contract. He was graduated from Oklahoma A & M College with a B.S. in chemical engineering in 1941.

D. V. MOSES

D. V. Moses, a technical superintendent at the Du Pont Co.'s Belle (W. Va.) plant, died recently. He was a native of Ames, Iowa, where in 1925 he received his Ph.D. in chemical engineering from Iowa State College after teaching there for six years. He then spent four years on water treatment with the Gover Corp. before joining the Du Pont Co. in 1929. He was regarded as an authority on water treatment, waste disposal, synthesis gas generation, and automatic analytical devices for gases. He was a member of the American Water Works Association and West Virginia Sewage and Disposal Association.

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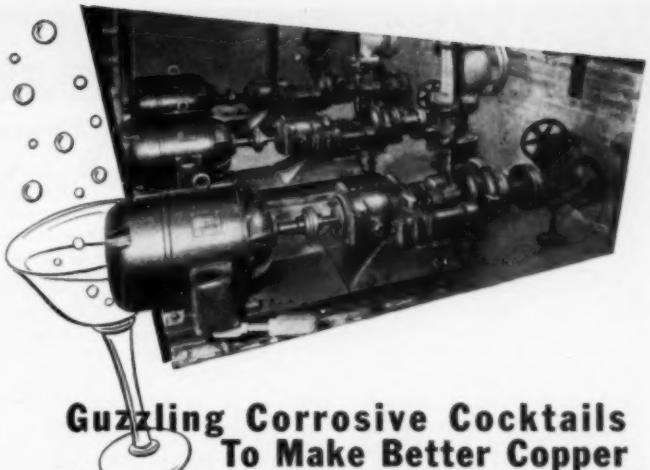

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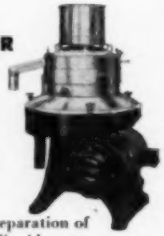
This disc type, continuous centrifuge utilizes the stratification principle which is most effective in concentrating a liquid or solid phase suspended in another liquid.

NOZLJECTOR

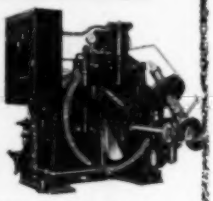


Solids are continuously discharged from the disc type bowl of this high efficiency centrifuge which is widely used for separation of two immiscible liquids containing solids, or for the concentration of solids in a liquid phase.

DV-2 CENTRIFUGE

Solids ranging from firm to slimes and sludges are handled by this versatile centrifuge, with continuous solids discharge. Available as a clarifier to remove solids from liquids; or a separator, to separate two immiscible liquids in the presence of solids.

SUPER-D-HYDRATOR



An extremely flexible, completely automatic centrifuge for recovery and dehydration of crystals. Efficiently handles slurries with 15 to 60% solids. Each step of feed, rinsing and drying can be individually controlled.

SUPER-D-CANTER

High capacity centrifuge for removal of solids from liquids . . . clarification of liquids . . . classification of solids. Available in two bowl types to accommodate wide range of solids consistencies.

Our Engineering Staff is always available to you. Feel free to call on their experience to analyze your needs and recommend the centrifuge that will do your job BEST.

SHARPLES

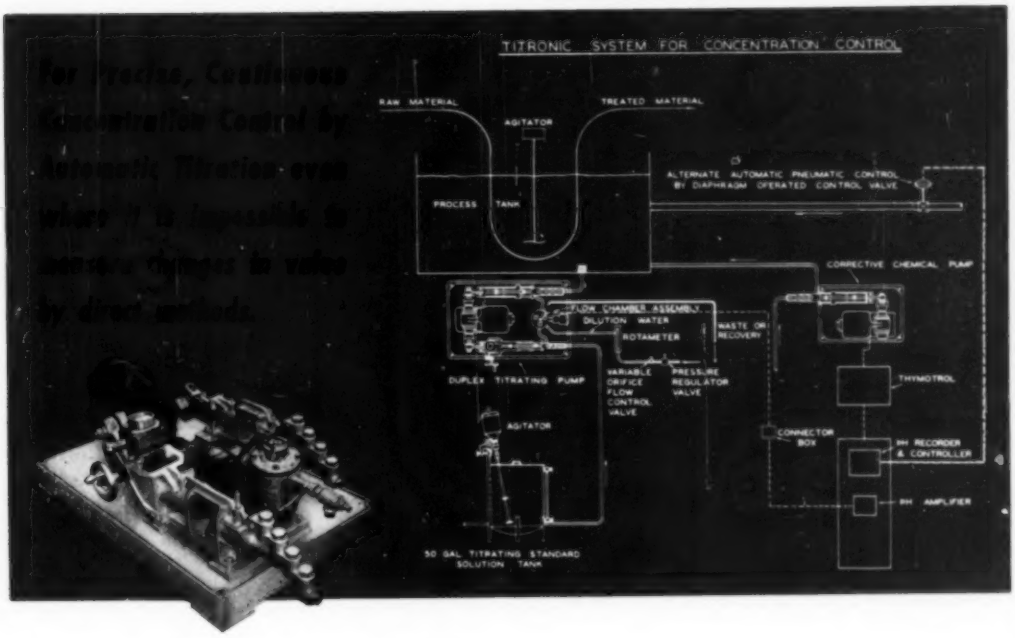
THE SHARPLES CORPORATION • 2300 WESTMORELAND STREET, PHILADELPHIA 40, PENNA.

NEW YORK • BOSTON • PITTSBURGH • CLEVELAND • DETROIT • CHICAGO • NEW ORLEANS • SEATTLE • LOS ANGELES • SAN FRANCISCO • HOUSTON

THE MILTON ROY

Automatic

"TITRONIC" SYSTEM



The new Milton Roy "Titronic" System utilizes one side of a Duplex Controlled Volume Pump to automatically and continuously titrate a sample from the liquid bath. The other side of the pump meters to the sample, in direct ratio and exact proportion, a fixed quantity of the titrating medium.

It makes it possible to control the strength of a caustic or acid bath where there is relatively little change in conductivity, pH value, specific gravity or other variable between the desired control limits.

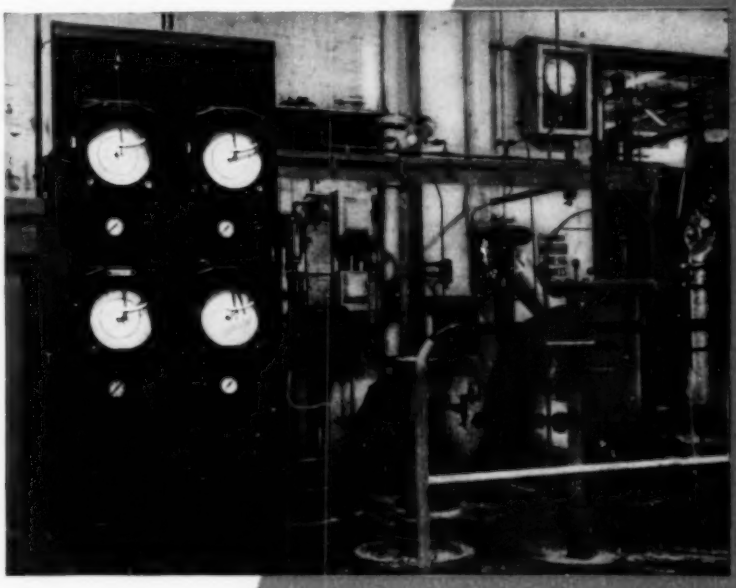
For example, if control is desired on a caustic bath a standard solution of phosphoric acid might be added to the caustic sample. The mixture of the two reagents would then give a resulting pH value on the steep portion of a curve that would directly indicate the strength of the bath. A meter measuring this pH value would automatically set the feed of strong caustic to maintain the standard strength of caustic required in the bath, increasing the feed in the event that the titration indicated a falling pH value below the control point, or decreasing the feed of caustic soda in the event that the titration indicated a bath of higher concentration than required.

Applications include maintaining strengths in acid or alkali baths, control of bleach baths or reducing baths by measurement of oxidation potential instead of pH value and others. Each application must be studied to determine the proper control point to be used according to the slope of the titration curve, desired buffer action, etc. Inquiries are invited.

MILTON Roy COMPANY

1379 E. MERMAID LANE, CHESTNUT HILL, PHILA. 18, PA.

Today's most flexible and dependable AUTOMATIC FLOW RATIO CONTROLLER



A panel of three F&P Ratio-Controllers blending wood pulp slurry with one additional recording-controller. Also shown are large-capacity RATOSLEEVE flow meters and control valves.

● WIDE RANGE OF APPLICATION

A choice of F&P primary elements is available to handle a range of flows as low as 4-40 cc/min liquid for pilot plant blending or treating-reagent additions to any measurable high capacity flow in closed or open channels.

● WIDE RANGE OF MEASUREMENT

The wide-range linear flow scales of F&P primary elements permit outstanding rangeability of flows under all conditions—not merely at or near design ratio.

● WIDE RANGE OF SETTING

Controlled flow can be in any desired ratio to "wild" or uncontrolled flow. Ratio selector scales are graduated from 15% to 600% (scale length is 7"). Pneumatic-set ratio is available for applications such as pH control. Vernier adjustment can be supplied for extreme precision of setting.

● COMPLETE RATIO-CONTROL SYSTEM

Full responsibility for the proper coordination and smooth performance of the various components of the system is vested in Fischer & Porter, manufacturer of the primary elements (FLOWRATOR meters, V/A Cell Kinetic manometers, and others)—secondary elements (RATOMATIC control instruments) — and VALVRATOR control valves.

Fischer & Porter offers a complete system for proportioning the flows of fluids (liquids, gases, slurries) in two or more pipe lines. The various elements utilize electrical, electronic or pneumatic transmission systems in any suitable combination. These systems can be readily tied in with machine speeds, positive displacement meters, metering pumps, or dry feeders.

WRITE FOR CATALOG 53

PROCESS CONTROL INSTRUMENTS

FLOWRATOR

FISCHER & PORTER COMPANY
HATBORO, PENNSYLVANIA, U.S.A.